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# DYESTUFF REPORTER
DEVOTED TO TEXTILE WET-PROCESSING

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AMERICAN
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Volume 46 June 17, 1957 Number 12

# EVALUATION OF WHITENING EFFICIENCY OF FLUORESCENT WHITENING AGENTS

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**Fluorescent whitening agents are dyes which absorb ultraviolet radiant energy and fluoresce blue. The fluorescense improves the appearance of textiles and paper in two ways; first, by shifting the color from yellow towards blue, and second, by increasing the lightness. The first of these effects is much greater in magnitude with fluorescent whiteners. These dyes may be evaluated by several methods: visual examination of the dyed cloth in daylight, visual examination under ultraviolet radiation, and examination by means of a fluorimeter. The advantages and disadvantages of each of these methods are discussed. If an ordinary fluorimeter is used to compare whiteners of different chemical composition, the results are meaningless unless a correction factor is used. A method of redesigning the fluorimeter so that it will not require a correction factor for this type of comparison is described.**

## INTRODUCTION

Most manufacturers and users of fluorescent whitening agents need an accurate method of evaluating these products for their whitening efficiency. This need is mainly felt in comparing whiteners of different types against each other, a task which constantly confronts the soap, textile, and paper manufacturers in their search for improved fluorescent whitening agents. This paper describes methods of evaluation now in use, and discusses their advantages and disadvantages. It also indicates improved methods which might be used for comparing whiteners of different types against each other.

## HOW DO FLUORESCENT WHITENING AGENTS WORK?

Before discussing methods of evaluating fluorescent whitening agents, let us review some of what is known about how these materials work. It might be well to start by considering the factors involved in the term "whiteness."

Figure 1 shows reflectance curves of a piece of 80 x 80 bleached cotton cloth four years old and of magnesium oxide, the latter being commonly accepted as a standard for whiteness. The curve for the bleached cotton differs from that for the magnesium oxide in two respects: it is lower, and it slopes upward from left to right. The lower reflectance means that the cotton is darker than magnesium oxide; the slope means that the cotton is yellower than magnesium oxide. Any method of improving the whiteness of the cotton should ideally both raise the curve and lessen the slope. The methods to be discussed are intended for use with bleached cloth, and are not substitutes for chemical bleaching.

One common method of whitening makes use of a blue dye or pigment;

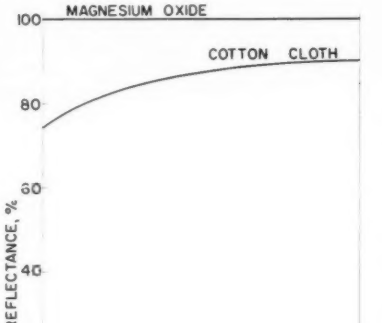


**Figure 1**

**Reflectance curves of magnesium oxide and old bleached cotton cloth**

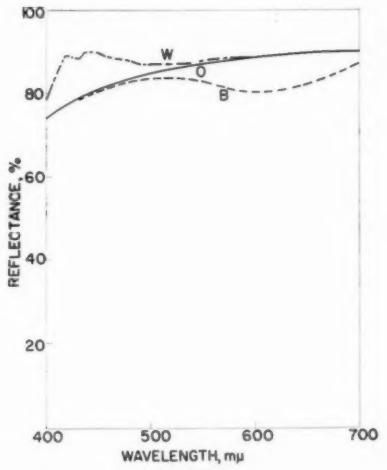


**Figure 2**

**Reflectance curves of old bleached cotton cloth, before and after treatment with laundry blue and fluorescent whitener**

O—Original cotton cloth
B—Same cloth treated with laundry blue
W—Same cloth treated with fluorescent whitening agent (apparent reflectance curve)

this is what the housewife does when she adds bluing to her wash. Figure 2 shows reflectance curves of the same piece of cloth as that used for Figure 1, both before and after treatment with laundry blue. Curve O represents the original cloth, and is identical with the lower curve in Figure 1. Curve B represents the same cloth treated with laundry blue. It can be seen that such treatment lessens the overall slope of the curve, but also lowers the curve. Thus, bluing decreases the yellowness, but also makes the cloth darker. In effect, it changes the color of the fabric to a light gray.

Another method of improving whiteness is by the use of a fluorescent whitening agent. These agents

are dyes which absorb ultraviolet radiant energy (always present in daylight) and re-emit this in the form of blue light. Figure 3 shows how this emitted light is distributed through the spectrum in the case of a red-shade whitener, Curve 1, and a green-shade whitener, Curve 2. The highest energy of emission occurs at a wavelength close to 440 millimicrons. There is relatively little emitted light above 500 millimicrons, which is about where the blue region ends. Note that the curve for the green-shade whitener is displaced to the right of that for the red-shade whitener. This shows that the former type gives off relatively more energy at the higher wavelengths, which accounts for its greener shade on cloth.

Since the emitted light from the whitener is added to the reflected light from the cloth, we can superimpose the emission curve of the whitener, shown in Figure 3, onto the reflectance curve of the cotton, shown in Figure 1, in order to find out what happens when a cotton cloth is treated with whitener. The method of doing this is described in the Appendix. Curve W in Figure 2 shows an "apparent reflectance curve," which illustrates the effect of dyeing the cloth with the red-shade whitener whose emission curve is shown in Figure 3. Note that the curve for the cotton treated with whitener not only has a lesser slope than the curve for the original cotton cloth, but is also higher than the latter. This shows that the fluorescent whitener not only takes away the yellow cast of the original cloth but also increases its lightness.

To make these effects more concrete, the curves shown in Figure 2 were calculated over to yellowness and lightness values (as explained in the Appendix). Figure 4 shows the colors of the original cloth, the cloth treated with laundry blue, the cloth treated wih fluorescent whitener, and magnesium oxide plotted on a diagram with yellowness as one axis and lightness as the other. The units are such that one unit of yellowness change is equal visually to one unit of lightness change. It is seen that use of laundry blue produces almost as much darkening of the cloth as it does decrease in yellowness. On the other hand, the fluorescent whitener lightens the cloth slightly while it decreases its yellowness. This shows graphically the great advantage of fluorescent whiteners over dyes of the laundry blue type.

The diagram also shows that the main effect of fluorescent whiteners is to decrease yellowness (or, what is the same thing, increase blueness).

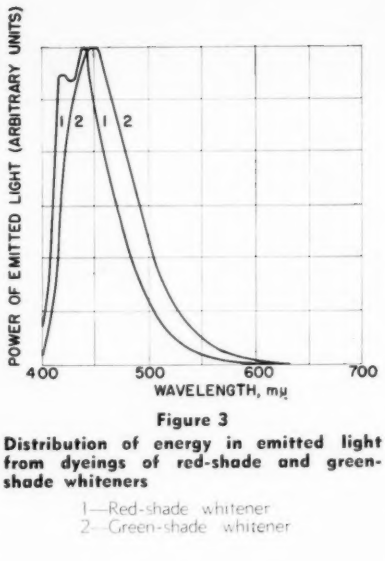


**Figure 3**

**Distribution of energy in emitted light from dyeings of red-shade and green-shade whiteners**

1—Red-shade whitener
2—Green-shade whitener

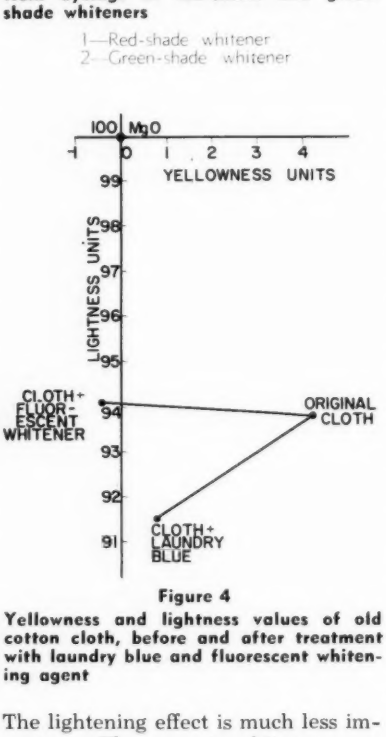


**Figure 4**

**Yellowness and lightness values of old cotton cloth, before and after treatment with laundry blue and fluorescent whitening agent**

The lightening effect is much less important. Fluorescent whiteners are sometimes called "brighteners." This would appear to be a misnomer, since brightness is associated with lightness. "Antiyellowers" would be a more descriptive term, although somewhat clumsy. Probably the more general term "whitener" is the best.

Depending on whether a red-shade whitener or a green-shade whitener is used, the treated cloth may appear reddish or greenish in hue. Which hue is preferred is a matter of personal choice. From the point of view of evaluating whiteners of different shades for their whitening efficiencies, it is best to disregard the final hue produced. We can do this by only considering the two fundamental effects mentioned above: the bluing effect, and the lightening effect. For this reason, the problem of hue preference will not be further considered in this article.

## AMOUNT OF FLUORESCENCE AS A MEASURE OF WHITENING EFFECT

It can be shown by theory that both the bluing and the lightening effects of a fluorescent whitener are proportional to the amount of fluorescence emitted by the whitener-treated material. Also, if two dyeings made with different whiteners have the same amount of fluorescence (as defined below), they appear equally white to the eye (*1*). Therefore, the evaluation of a fluorescent whitener consists in determining the amount of its fluorescence when dyed on cloth. To compare two different fluorescent whiteners, A and B, for their whitening efficiency, one determines the concentration of Whitener B which gives the same amount of fluorescence as a fixed concentration of Whitener A. The ratio of these two concentrations is then a direct measure of the relative whitening efficiencies of Whiteners A and B.

However, we are immediately faced with a fundamental problem: How do we define "amount of fluorescence?" That this is not an easy term to define may best be illustrated by an experiment: A dyeing on cotton of the red-shade whitener whose emission curve is shown in Figure 3 was made at a concentration of 0.0060% on the weight of the cloth. Dyeings on cotton of the green-shade whitener whose curve is shown in Figure 3 were made at varying concentrations. These dyeings were compared visually under ultraviolet illumination to the dyeing of the red-shade whitener, and judged for amount of fluorescence. It was found that a 0.0066% dyeing of the green-shade whitener matched the 0.0060% dyeing of the red-shade whitener for amount of fluorescence. The same dyeings were then read on a fluorimeter with the same light source as was used in the visual examination. It was found that a 0.0142% dyeing of the green-shade whitener matched the 0.0060% dyeing of the red-shade whitener for amount of fluorscence. Thus, although both methods evaluated amount of fluorescence, different results were obtained depending on how the sample was examined.

This discrepancy is easy to explain. Neither the human eye nor the fluorimeter is equally sensitive to all wavelengths of light. Figure 5 shows the relative sensitivity of the human eye

to stimulation by different wavelengths of light. A similar curve is also shown for the fluorimeter used in the experiment described above. Note that the human eye has no sensitivity to ultraviolet radiation and very little sensitivity to violet (400-450 millimicrons). The sensitivity of the eye goes up in the blue (450-500 millimicrons) and reaches its maximum in the green (500-570 millimicrons). On the other hand, the fluorimeter is very sensitive in the low wave-length regions of violet and blue.

Now consider the emission curves of Figure 3. The green-shade whitener emits more light at higher wavelengths than does the red-shade whitener. Since the eye is more sensitive to these higher wavelengths, the green-shade whitener is favored in the visual examination. On the other hand, since the fluorimeter is more sensitive to the lower wavelengths, the red-shade whitener is favored in the fluorimeter. Therefore, much less green-shade whitener has to be applied to the cloth to match the 0.0060% dyeing of the red-shade whitener when the comparison is made visually than when the comparison is made by fluorimeter.

Now it may seem that the visual response is the only one with which we are concerned. After all, the housewife looks at her sheets and pillow cases by eye; she does not measure them in a fluorimeter. However, the situation is not quite so simple. The visual response curve shown in Figure 5 shows the relative effect of different wavelengths of light in stimulating the eye. The more the eye is stimulated, the more light it sees. Therefore, this curve can be correctly used to predict the effect of different whiteners in making the cloth *lighter*. But we indicated previously that fluorescent whitening agents have two effects, a lightening effect and a bluing effect, of which the latter is of much greater magnitude and thus much more important. The visual sensitivity curve tells nothing about the sensitivity of the eye to color changes, and therefore does not indicate what the bluing effect would be under daylight examination.

As we have seen, both the eye and the fluorimeter are not equally sensitive to all wavelengths of light. Suppose we had a viewing device which *would* be equally sensitive to all wavelengths. Such a device would measure all the fluorescence from a dyeing of whitener without favoring one wavelength over another, and would therefore give a reading for amount of fluorescence which would

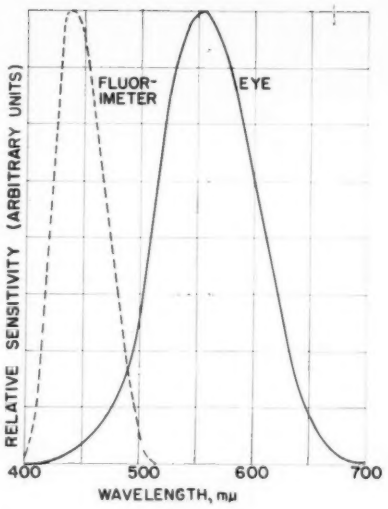


**Figure 5**

**Relative sensitivity of human eye and of fluorimeter to different wavelengths of light**

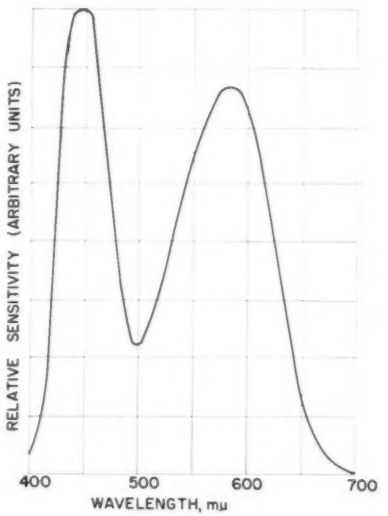


**Figure 6**

**Relative sensitivity of viewing device which would give theoretically correct indication of whitening power**

be more nearly correct from the physicist's point of view. Would this kind of fluorescence measurement be a good measure of whitening power? It would not, and this can easily be illustrated by imagining a hypothetical "fluorescent whitener" whose emission curve would be displaced 100 millimicrons to the left of those shown in Figure 3. Such a dye would emit in the ultraviolet. But the human eye cannot see ultraviolet radiation. Therefore, such a dye would be no good as a whitener, although it would register a high reading on our viewing device which is not selective in wavelength.

Therefore, if we are to determine whitening power by reading amount of fluorescence, we need some sort of viewing device which would weight the different wavelengths of fluorescence in such a way as to favor those wavelengths which contribute most to the whitening effect of the dye. Since the whitening effect is visual, we need a response curve which is tied up with the visual mechanism; however, we cannot use a curve which is restricted to lightness effect alone, like that shown in Figure 5.

Theoretical considerations (*1*) show that the response curve desired is the one shown in Figure 6. If dyeings of fluorescent whiteners are irradiated with ultraviolet and examined by some device having the relative sensitivity to light at different wavelengths shown in this curve, an accurate measure of the whitening effect in daylight (comprising both bluing effect and lightening effect) is obtained, regardless of the type of whitener. This curve is the sum of the tristimulus functions, $x+y+z$. We can therefore define "amount of fluorescence," or, more accurately, "effective fluorescence," as the measured fluorescence under ultraviolet illumination, this fluorescence to be sensed by a device with the response curve shown. Thus defined, "effective fluorescence" is a direct measure of daylight whitening power for any fluorescent whitener.

It should, perhaps, be emphasized that this strict definition becomes necessary only when different types of whiteners are being compared. For evaluations with only one particular whitener, such as might be undertaken in determining the effect of concentration on fluorescence, any viewing device would be adequate. The ordinary type of fluorimeter is particularly useful in such study. It is, however, well known that a fluorimeter cannot be used for comparing two different whiteners; this could be done if the fluorimeter had the response curve shown in Figure 6, and if certain other modifications were made. This will be described in greater detail later in the article.

## METHODS OF EVALUATING FLUORESCENT WHITENING AGENTS FOR THEIR WHITENING POWER

With this much of a background, let us now examine critically some of the methods in use for evaluating fluorescent whitening agents for their whitening power. Each method has

certain advantages and disadvantages, which we will be able to understand in the light of the previous discussion.

It should be mentioned at the outset that most of the methods commonly used to measure whiteness of nonfluorescent whites cannot be used with fluorescent whiteners. Such instruments as the GE Brightness Meter, the General Electric Recording Spectrophotometer, and most of the three-filter colorimeters on the market give completely misleading results. Entirely new methods are required with these substances, because of their fluorescent properties.

VISUAL EXAMINATION IN DAYLIGHT———This is the most accurate method of evaluation, since it gives directly the results required. The housewife judges a fluorescent whitener by examining her wash in daylight as it is hanging on the line. Although she does not make a detailed analysis of her visual sensations, in effect she integrates the bluing effect and the lightening effect to obtain an overall estimate of whiteness.

However, this method has two major disadvantages. The first is that although this is the most accurate method, it is certainly the least precise. Accuracy is a measure of how closely the results approach the correct value, and the method is accurate because it gives directly the correct value. But precision is a measure of how reproducible duplicate determinations are, and the method fails in this respect because slight differences are difficult to see. For example, in dyeings with the same whitener, the eye can barely distinguish a 10% change in strength by daylight examination. This would make the method unsuited to control of dye quality or studies of build-up, lightfastness and the like. Daylight evaluation becomes even more uncertain when whiteners of different shade are compared against each other, since the shade difference confuses the estimation of whiteness.

The second disadvantage relates to a standard light source which duplicates daylight in spectral quality. Daylight varies in spectral quality not only from day to day but from minute to minute. If any reproducible work is to be done, it is necessary to specify an artificial light source which would approximate daylight, would be unvarying in quality, and could be exactly duplicated from laboratory to laboratory. The Inter-Society Color Council has set up Subcommittee for Problem 18, Colorimetry of Fluorescent Materials, which is actively working on this problem. It is hoped that a standard light source will be available in the near future.

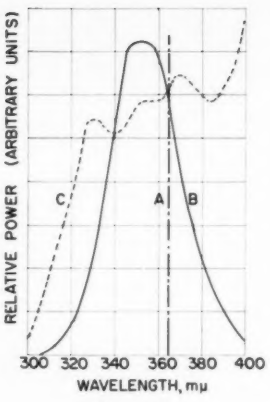


**Figure 7**

**Distribution of energy from different light sources**

A—AH-5 mercury lamp with Corning No. 5874 filter
B—T12 black light lamp with integral filter
C—Daylight (Middleton)

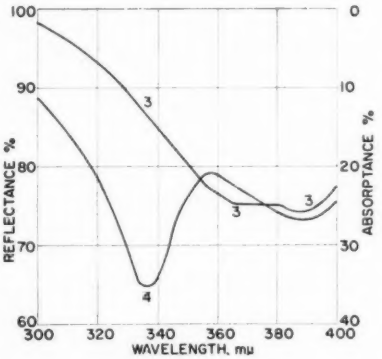


**Figure 8**

**Reflectance curves of two fluorescent whitening agents**

VISUAL EXAMINATION UNDER ULTRAVIOLET ILLUMINATION———The remaining methods to be considered all evaluate amount of fluorescence. The simplest method of doing this is to look at the dyeings under ultraviolet radiation, and judge the fluorescence visually.

Because of its simplicity, this is an excellent method, but unfortunately its use is limited to evaluation of dyeings made with the same fluorescent whitener, for reasons to be discussed in the next paragraph. However, this method can be used in examining dyeings made with different whiteners if one is only interested in shade differences, for example, to judge whether a given sample is redder or greener in hue than a standard sample.

For checking dyeings with the same whitener for amount of fluorescence, this method is more precise than evaluation in daylight; a strength difference of 5% may readily be seen. Therefore, the method is suitable for studies on build up, lightfastness, variations of fluorescence with concentration of whitener, and similar problems.

The major disadvantage of this method is that it cannot be used for comparing dyeings made with different whiteners for their amounts of fluorescence, except where very rough evaluations are needed. There are two reasons for this. First, as was discussed previously, the response of the eye does not have the correct characteristics for judging the whitening effect in daylight. This was explained in connection with the discussion of Figures 3, 5, and 6. Second, the ultraviolet sources commonly in use for examining whitener dyeings do not agree in spectral energy distribution with the ultraviolet component of daylight. That this may produce an error can be illustrated by the following experiment:

A 0.0060% dyeing on the weight of the fabric of a fluorescent whitening agent, designated Whitener 3, was made on cotton. A series of dyeings at varying concentrations of another fluorescent whitener, designated Whitener 4, was made on cotton. These dyeings were then examined visually under ultraviolet illumination from two different light sources. One was an AH-5 mercury lamp with Corning No. 5874 filter to cut out the visual light. Almost all of the ultraviolet energy from this light source is concentrated at the single wavelength of 365 millimicrons. The other was a 40-watt T12 black light lamp with integral filter. This lamp gives off radiation over a range of wavelengths in the ultraviolet. The spectral energy distributions from both lamps are shown in Figure 7, where A refers to the AH-5 lamp and B to the T12 lamp. Comparisons were made under each lamp to determine which dyeing of Whitener 4 most closely matched in amount of fluorescence the 0.0060% dyeing of Whitener 3. Under the AH-5 lamp it was found to be the 0.0090% dyeing of Whitener 4 but under the T12 lamp it was the 0.0050% dyeing of Whitener 4. Thus, Whitener 4 showed greater fluorescence under the T12 lamp than it did under the AH-5 lamp, as compared to Whitener 3.

These results are easily explained. Figure 8 shows reflectance curves of Whiteners 3 and 4 in the ultraviolet region of the spectrum (*1*). The vertical scales are given both in reflectance and absorptance, where percent absorptance equals 100 minus percent

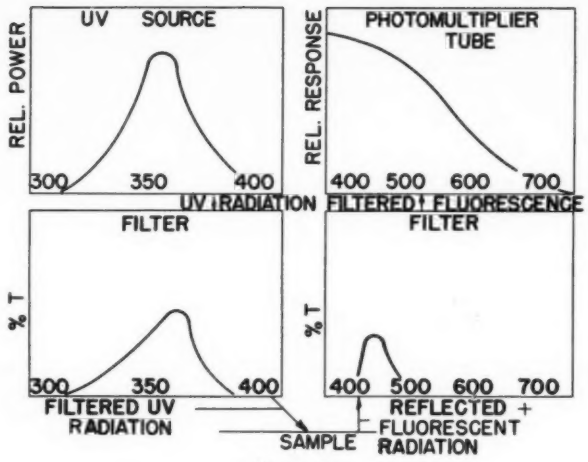


**Figure 9**
**Block diagram of modified GM fluorimeter**

reflectance. The higher the percentage of ultraviolet radiation absorbed, the greater the fluorescence, other things being equal. Now the AH-5 lamp only gives off radiation substantially at 365 millimicrons, as is shown in Figure 7. On the other hand, the T12 lamp gives off radiation both above and below 360 millimicrons. Whereas Whiteners 3 and 4 have close to the same absorptance values at 360 millimicrons and above, Whitener 4 absorbs much more strongly in the region of 340 millimicrons than does Whitener 3. Therefore, Whitener 4 is more favored under the T12 lamp than under the AH-5 lamp, compared to Whitener 3. This simple experiment shows that the distribution of energy in the ultraviolet source is of great importance in judging or measuring fluorescence.

Neither of these lamps has the same ultraviolet energy distribution as daylight. The ultraviolet energy distribution of daylight is also shown in Figure 7 as curve C *(2)*. We can see that if we had a fluorescent whitener which absorbed strongly in the region of 390 millimicrons, where daylight has quite a bit of energy but both of the lamps have little or no energy, such a whitener would be penalized under the ultraviolet lamp as compared with daylight. It is generally true that even if the problem of finding a viewing device with the correct spectral sensitivity mentioned earlier were solved, misleading results would still be obtained unless the ultraviolet light source duplicated daylight in energy distribution.

USE OF FLUORIMETER——Fluorimeters are commonly used to determine fluorescence intensity of dyeings with fluorescent whitening agents.[1] Quite a few such instruments have been developed, but they are all based on the same principle. Figure 9 shows a block diagram of the fluorimeter which is used at Cynamid at the present time.[2]

Radiation from a black light ultraviolet lamp passes through a filter (Corning No. 5860 Violet Ultra filter glass), the function of which is to cut out all visible light and pass only the ultraviolet. This ultraviolet radiation then strikes the sample, giving rise to fluorescent light in the visual region. However, some ultraviolet radiation is reflected by the sample, and this must be removed by another filter before the fluorescence is measured by the photomultiplier tube. The latter filter is a Wratten No. 47A, which cuts out ultraviolet and passes the blue fluorescence. Our particular fluorimeter has a series of range switches which makes it possible to read any degree of fluorescence from very weak to very strong. Readings are reproducible, and have a high degree of precision.

The fluorimeter provides the most precise method of determining fluorescence; in a series with any one whitener a strength difference of 1.5% can easily be detected. This makes the fluorimeter ideally suited to exact studies of the type mentioned above in the section on visual examination under ultraviolet illumination; it is also quite useful for quality control of shipments of fluorescent whitener. However, as in the case of the visual method, it cannot be used directly for comparing two different types of whitener, and exactly for the same reasons. It can be seen from Figure 9 that the energy distribution of the ultraviolet lamp, particularly after modification by the transmission characteristics of the filter, is quite different from that shown for daylight in Figure 7. Furthermore, the response curve of the photomultiplier tube, as modified by the transmission curve of the filter in front of it, is considerably different from the theoretically correct response curve shown in Figure 6. These limitations of the fluorimeter are not always recognized, and misleading results have been obtained when whiteners of different types were compared against each other.

USE OF FLUORIMETER WITH CORRECTION FACTOR——It is possible to apply a correction factor to readings obtained on a fluorimeter so that fluorescent whiteners of different types may be compared against each other *(1)*. This factor is really the product of two separate factors; the first corrects for the discrepancy between the energy distribution of the light source used in the fluorimeter and the energy distribution of ultraviolet radiation in daylight, and the second corrects for the discrepancy between the response curve of the photomultiplier tube plus filter and the theoretically desired response curve. These factors are different for each pair of whiteners compared, and must be recalculated for each new case. However, once the calculation is made, the factor can be used indefinitely for the same pair of whiteners. The method of calculating these factors is given in the reference cited at the beginning of this paragraph.

After the correction factor for a particular pair of whiteners is calculated, the determination of the relative whitening efficiency of these whiteners is made fairly simply. The method is illustrated in Figure 10. In this particular experiment, a 0.0060% dyeing on the weight of the cloth of a particular whitener, which we will designate Whitener P, was made. This dyeing was read in the fluorimeter, and gave a value of 75.5. A series of dyeings of another whitener, which we will designate Whitener Q, was made at concentrations ranging from 0.0080% to 0.0110% on the weight of the cloth. These were read on the fluorimeter; and the readings

[1] For a list of references to the use of the fluorimeter, see Ref *(1)*.

[2] This is a modification of the G-M fluorimeter, originally developed for determining uranium. The changes comprised the insertion of the filter after the ultraviolet lamp, the substitution of a different filter before the photomultiplier tube, and the use of a new sample drawer adapted for use with cloth samples. Both the original and modified instruments are available from Engineering Equipment Company, P O Box 1506, Boynton Beach, Fla.

were multiplied by the correction factor which converts such readings to the same basis of effective fluorescence as for Whitener P. The corrected readings were plotted against concentration, and the best straight line (by least squares) was drawn through the points, as shown in the figure. It is seen that a 0.00886% dyeing of Whitener Q would give the same fluorescence as did the 0.0060% dyeing of Whitener P. Therefore, Whitener Q is 0.0060/0.00886 as effective as Whitener P; this amounts to a relative efficiency of 68%. It should be mentioned that the curve of fluorescence against concentration is not a straight line over a wider concentration range; however, the straight line is the most convenient plot to use over a limited concentration range.

This method has all the advantages of the best methods considered so far. The precision obtainable is, of course, equal to the precision obtained by the use of the fluorimeter without the correction factor. The accuracy approaches that obtained by direct examination in daylight; however, some error may be introduced in the theoretical assumptions made in calculating the correction factors, as well as in the data used for this calculation. In general, this is probably the most satisfactory method available for comparing whiteners of different types for their whitening power.

The disadvantage of this method lies in the specialized apparatus necessary to obtain the data from which the factors are calculated. The data needed are the emission curves of both whiteners, such as those shown in Figure 3; the ultraviolet reflectance curves of both whiteners, such as those shown in Figure 8; the energy distribution curve of the fluorimeter light source and the transmission of the filter in front of it, such as shown in Figure 9; also the response curve of the fluorimeter such as that shown in Figure 5. This requires specialized equipment which is not available in many laboratories. Once the data are obtained, the correction factor may be calculated in about one hour.

USE OF SPECIALLY DESIGNED FLUORIMETER——The ideal way to compare two different fluorescent whitening agents for their whitening power would be to use a fluorimeter which would give direct readings in effective fluorescence, and which would require no calculation. We do not as yet have such an instrument, but plan to build one.

Figure 11 shows a block diagram

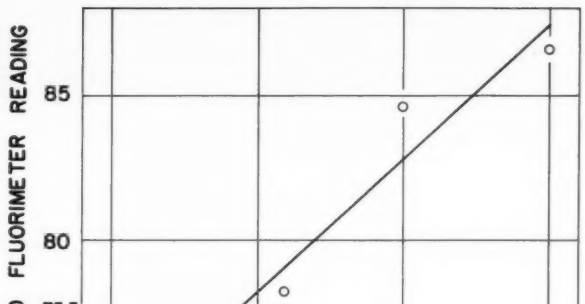


Figure 10

Method of calculating relative efficiency of two fluorescent whitening agents

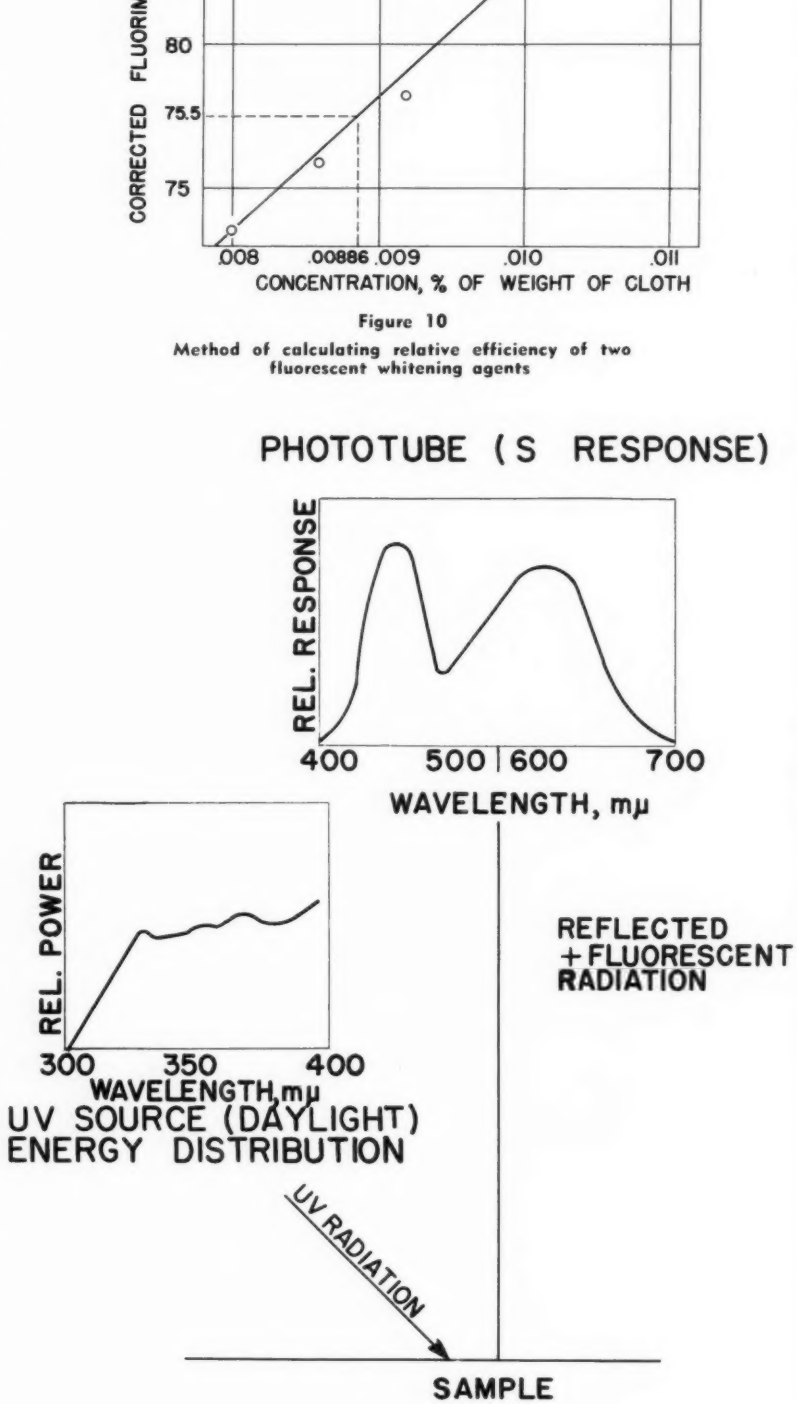


Figure 11

Block diagram of ideal fluorimeter which would not require a correction factor for comparing whiteners of different types

of an instrument of this type. The light source has an energy distribution identical to that of the ultraviolet component of some standard phase of daylight, and the photosensitive device has a response curve identical to the theoretically desired one. Such a fluorimeter would combine the accuracy and precision of the best of the presently available methods, with the further advantage of extreme simplicity. The difficulty would be to find the proper light source and the proper filter-phototube combination.

Alternatively, by using filters which, in combination with the phototube, would give readings of the separate tristimulus values, X, Y, and Z, of the emitted light, the hue as well as the whitening power could be determined. The separate tristimulus values could be easily converted to a quantity representing hue. The sum of the tristimulus values would be a measure of the effective fluorescence intensity or whitening power. This is because the theoretically desired response curve of the phototube, shown in Figure 6, actually represents the sum of the tristimulus functions $\bar{x}+\bar{y}+\bar{z}$, as was mentioned previously.

RESULTS BY METHODS DESCRIBED——To show the results obtainable by the various methods described (except the last, for which a fluorimeter has not yet been built), six different fluorescent whitening agents (designated B to G, inclusive) were compared against a seventh (designated A). A dyeing of Whitener A on cotton was made at a concentration of 0.0060% on the weight of the cloth. Dyeings of each of the other whiteners were made on cotton at varying concentrations. The concentration of each whitener having the same effective fluorescence as 0.0060% of Whitener A was determined by each method. Results are shown in Table I.

**TABLE I**

**Comparison of Whiteners for Whitening Power by Different Methods**

Concentration Required to Give Same Whitening Power as 0.0060% of Whitener A

| Whitener | Visual examination in daylight[a] | Visual examination under ultraviolet light | Use of fluorimeter | Use of fluorimeter + corr factor |
|---|---|---|---|---|
| B | .0096 | .0090 | .0142 | .0106 |
| C | .0096 | .0090 | .0141 | .0096 |
| D | .0092 | .0070 | .0092 | .0086 |
| E | .0075 | .0073 | .0077 | .0069 |
| F | .0058 | .0060 | .0062 | .0059 |
| G | .0055 | .0050 | .0049 | .0048 |

[a] Visual examination in daylight was made directly in terms of whitening power; examination by the other methods listed was made in terms of amount of fluorescence as an indication of whitening power.

**TABLE II**

**Comparison of Methods for Evaluating Fluorescent Whitening Agents**

| Method | Distinguishable strength difference[a] | Advantages | Disadvantages |
|---|---|---|---|
| Visual examination in daylight | 10% | Simplest and most direct method | 1) Poor precision, especially in comparing whiteners of different shade<br>2) Great variation in daylight from minute to minute makes results unreliable |
| Visual examination under ultraviolet illumination | 5% | 1) Very simple and convenient<br>2) Fairly good reproducibility for work with a single whitener | Poor results in comparing whiteners of different types |
| Use of fluorimeter | 1½% | 1) Fairly simple and convenient<br>2) Very good reproducibility for work with a single whitener | Poor results in comparing whiteners of different types |
| Use of fluorimeter with correction factor | 1½% | 1) Very good reproducibility for work with a single whitener<br>2) Good results in comparing whiteners of different types | Specialized equipment needed to obtain data required for calculation of correction factor |
| Use of redesigned fluorimeter | 1½% | 1) Simple and convenient<br>2) Very good reproducibility for work with a single whitener<br>3) Good results in comparing whiteners of different types | Difficult to design such a fluorimeter |

[a] In dyeings with same whitener.

The results in Table I show that the use of fluorimeter plus correction factor gives fairly accurate results, based on comparison with results obtained by visual examination in daylight. It must be remembered that visual examination in daylight is rather uncertain, particularly where whiteners of different shades are being compared. The use of a fluorimeter without correction factor gives seriously inaccurate results for Whiteners B and C, and visual examination under ultraviolet light gives inaccurate results for Whitener D.

## SUMMARY

In summary, fluorescent whitening agents are dyes which absorb ultraviolet radiation and emit this energy in the form of blue light. The emitted light serves two functions: the first is to counteract the yellowness of the substrate, and the second is to make the substrate lighter. Both of these effects contribute to improving the whiteness of the substrate. However, the decrease in yellowness is much greater than the increase in lightness. In contrast with fluorescent whitening agents, dyes and pigments of the laundry blue type make the substrate darker while removing the yellowness.

A convenient way of evaluating fluorescent whiteners for their whitening power is to measure their "effective fluorescence" (as defined in this article), since two dyeings with equal effective fluorescence appear equally white to the eye, even though two entirely different whiteners may be involved. Of course, dyeings may be also evaluated by direct examination in daylight, but this is unsatisfactory for several reasons. There are several methods available or in the process of development for evaluating effective fluorescence. Table II presents a summary of the characteristics, advantages and disadvantages of each of the methods.

The last method listed in Table II is at the drawing board stage at present, but we hope that a fluorimeter having the desired characteristics will be designed shortly. Work is in progress on this instrument in our laboratory.

## APPENDIX

CALCULATION OF "APPARENT REFLECTANCE CURVE" OF COTTON CLOTH TREATED WITH FLUORESCENT WHITENER, SHOWN IN FIGURE 2———A spectrophotometer cannot be used for measuring fluorescent materials. Although there are ways of determining the energy distribution from fluorescent samples, it was considered simpler for the purposes of this article to obtain the energy distribution of cotton cloth treated with fluorescent whitening agents by calculation.

Since the emitted light arising from fluorescence combines additively with the reflected light from the cloth, it is only necessary to add the energies of emission and reflection at each wavelength. (The yellowing effect of the dye on the reflected light, arising from some absorption in the blue, is disregarded as being small compared to the effect of the fluorescence, for ordinary concentrations of dye.) The reflectance curve was first multiplied through at each wavelength by $E_C$ (the energy distribution of Source C), in order to obtain the energy distribution of the reflected light, under the assumption that the sample is illuminated by Source C. This energy was added, at each wavelength, to the energy arising from the emitted light for the red-shade whitener (Figure 3). The resulting energies were divided, at each wavelength, by $E_C$ to obtain an "apparent reflectance curve" of the cotton cloth treated with fluorescent whitener. Care was taken in adding the fluorescence energies to the reflectance energies to use that amount of fluorescence energy which would produce a fairly linear apparent reflectance curve. This corresponds to adding just enough fluorescent whitener to the cloth to counteract the yellow tinge and obtain a neutral hue.

CALCULATION OF YELLOWNESS AND LIGHTNESS VALUES SHOWN IN FIGURE 4———This calculation was made in terms of Adams chromatic-value space. The reflectance (and "apparent reflectance") curves shown in Figure 2 were integrated for Source C illumination, and the tristimulus values were converted to the modified Adams system described by Glasser and Troy (3). L was taken as a measure of lightness, and b was taken as a measure of yellowness.


## ACKNOWLEDGMENT

The writer is indebted to F G Villaume and C B Villaume for their invaluable advice on brightener evaluation; also, to C B Villaume and assistants for making the dyeings and the visual evaluations.


## REFERENCES


(1) Eugene Allen, Submitted for publication in *J Opt Soc Am.*

(2) W E K Middleton, unpublished report to the Inter-Society Color Council Subcommittee for Problem 18, Colorimetry of Fluorescent Materials, dated March 22, 1954. A somewhat smaller reproduction of this curve is given in *J Opt Soc Am* **44**, 797 (1954), curve for energy distribution of an overcast sky over a hypothetical urban area.

(3) L G Glasser and D J Troy, *J Opt Soc Am* **42**, 657 (1952).


# Abstracts

### Identification of Synthetic Fibers by Micro Fusion Methods

Grabar, D G and Haessly, R, *Anal Chem* **28**, 1586-9, October, 1956

The technical literature abounds in descriptions of various techniques for the identification of textile fibers. These methods are based principally on microscopical observations of morphological characteristics visible in cross-section and longitudinal views, together with supplementary tests such as staining, solubility, and refractive index. Although in favorable cases each of these techniques may afford positive identification in itself, in the majority of cases one or more of the tests must be used to confirm the results obtained by another.

The scheme for the identification of synthetic fibers by the use of micro fusion methods, described by the authors, is based on the melting point of the fiber, the eutectic temperature of the fiber with *p*-nitrophenol as a reference compound, and the characteristic behavior observed during the heating and cooling of the fibers. Observations are made using a hot stage on a polarizing microscope.

This method is self-sufficient in most instances, but in some cases also must be confirmed by one or more of the other tests. However, it presents advantages over previously described techniques in being more widely applicable to both dyed and undyed, bright and dull, and filament and staple fibers. It is also faster and more positive, according to the authors, in distinguishing between chemically similar fibers, eg, nylon type 6 and nylon type 66. Although the fibers used in the test are destroyed, only very small samples are required.

A table is given, showing the melting point and/or the eutectic melting point in degrees C for all of the common synthetic fibers. The temperatures given for melting points of the fibers themselves are the ranges from the first sign of melting to the disappearance of the last trace of birefringence. Eutectic melting points are the temperatures at which definite melting is first detected.

This technique, which is described in great detail, is said to be applicable to staple and filament yarns and to bright and delustered fibers. With dyed fibers, polarization colors are obscured, but otherwise there is usually no interference.

Five references to the literature are cited.—WHC

### Glass-fiber Fabrics Find More Industrial Uses

Anon, *Textile World*, **107**, 120-1, April, 1957

Glass-fiber fabrics are being manufactured for many industrial uses, such as parts for automobiles, airplanes, trains, electronic devices, electrical equipment, and many others.

Properties that make the fibers of great value in industrial fabrics are high heat resistance, low moisture absorption, high specific gravity, good thermal conductivity, high tensile strength, durability, dimensional stability, acid and alkali resistance, and nonburning.

The first major use of glass-fiber textiles was in electrical equipment, wire and cable. For this purpose, glass fabrics are laminated with resins, glass-fiber yarns are braided with asbestos fibers, and glass tapes and braids are treated with rubber, varnish, silicones and other insulating compounds.

Glass cloth reinforced with plastics is used for aircraft parts, boat hulls, and structural building panels. Coated with rubber, it is used for convertible automobile tops, upholstery, window shades, etc. These coated fabrics have high tear strength and dimensional stability, and are

*(continued on page 462)*

Historical Notes (No. 13)—

# LO-KAO
# The Story of Chinese Green

SIDNEY M EDELSTEIN
*Dexter Chemical Corporation*
*New York, N Y*

This is the thirteenth in a series of historical articles which the textile chemist and colorist will find of interest.

The author, a prominent science historian, is the founder of an outstanding collection of great works, documents and letters in the history of chemistry and related sciences.

Articles and illustrations in this series are from the author's library.

THE unusual fact of history is coming into its own today. The more obscure the fact, the better. Witness the popularity of television quiz shows with emphasis on information from the past. Now, in this country, there is an appreciation for the forgotten men and forgotten deeds of the past. The history of dyeing also contains its forgotten men and events.

We have just finished celebrating the 100th anniversary of the discovery of the dyestuff Mauve. Who, however, knows of the discovery of Lo-Kao, the first true green dyestuff, which was made at almost the same time? Perkin was rightfully honored, but does anyone remember the Frenchman, Persoz, and the American, Forbes? The story of Lo-Kao certainly deserves retelling today.

The background setting is most important. Of all the colors, green is certainly the one that man has seen in most profusion from the earliest times. The green of the fields, the forests, and the green of the sea, have constantly been before his eyes.

From early times, man has had dyestuffs at hand for coloring his fabrics various shades of blue of excellent fastness. These blues were obtained by dyeing in the vat with woad, pastel, or indigo. Also, from the earliest times, man could dye brilliant and fast reds with kermes insect, cochineal or madder together with aluminum mordants. Reasonably fast and brilliant yellows could be obtained from a number of natural dyestuffs, such as weld and persian berries on alum mordants.

Green, however, presented a completely different story. To be sure, many plants would provide green juices which would give greenish stains on fabrics, but in comparison with the colors obtained with woad or indigo, cochineal, weld or Persian berries, these greens were practically worthless. For many centuries the only answer for obtaining satisfactorily dyed greens was (*1*):—

> 'Dye the fabric a satisfactory blue and then dye on top of the fabric a satisfactory yellow and thus obtain a satisfactory green.'

To the modern dyer, the matter of using a blue and a yellow dyestuff to obtain a green doesn't seem to present so great a problem. He can certainly choose a yellow and a blue of the same class and his only difficulty might be slight differences in rates of exhaustion. In the past, however, blues were applied to fibers by one type of dyeing method, namely, the reduction bath, whereas the yellows were applied in a hot or boiling bath on an alum mordant. This meant, therefore, that satisfactory greens were obtained by making two completely different dyeings. In other words, dyeing one yard of a fabric green required as much work and cost as much as dyeing two yards of the same fabric blue, or yellow, or red.

While the production of good greens on wool and cotton presented the expected difficulties and extra cost, in the case of silk, greens were especially exasperating. Macquer the famous French authority on silk dyeing of the 18th century had this to say about green on silk (*2*):—

> "This colour, composed of blue and yellow, is with difficulty produced on silk, because the blue vat is liable to spot and to give a pasty colour, an inconvenience more perceptible in green than blue."

Long after the time of Macquer, the difficulty in dyeing reasonably good greens on silk continued. With the development of the great silk industry of France, the increasing demand for greens was enough to make every dyer, silk exporter or merchant see "red." The status of the "green" problem in general at the end of the 18th century was summed up by Berthollet in his "Elements of the Art of Dyeing" (*3*):—

> "Many different plants are capable of affording green colours such as the field broom-grass (coquile noire) bromus secalinis, the green berries of the berry-bearing alder, wild chervil or cow weed, purple or honeysuckle clover, common reed; but these colours are not permanent.
>
> "It is by the admixture of blue and yellow that the dyers make green, which is distinguished into a great number of different shades; it requires address and experience to obtain this color uniform and without spots, particularly in the light shades.
>
> "Green may be produced by beginning either with the yellow or the blue dye; but the first method is attended with some inconveniences for the blue soils the linen and a part of the yellow being dissolved in the vat changes and makes it green; the second method is therefore preferred."

In spite of these problems in dyeing greens, one dyer of the last century was able to rhapsodize (*4*):—

> "Green is the medial color of the secondaries being a compound of yellow and blue in the proportions of three of yellow to eight of blue. Its melodizing tones being these two primaries and its contrasting color red —Green is nature's favorite color prevailing to a far greater extent in the vegetable kingdom than any other— of all decided colors, green may be used with most freedom in manufacture, in carpets, especially. It should almost always preponderate."

The difficulties in dyeing green, the high expense, and the great demand for the color naturally caused a continuing search for a green dyestuff which in itself would dye satisfactory greens on textiles. These searches were not made by the synthetic organic chemist, for up until Perkin's great discovery the idea of actually manufacturing a dyestuff by pure chemical means probably did not exist. But where could such a dyestuff be found? Obviously, only in some unknown plant or insect from some strange land. The discoverer of this green dyestuff therefore would have to be a botanist, a naturalist or explorer. There are at least 18 written records of supposed discoveries of such a dyestuff. Most of these, however, have proved to have been either fakes or at least commercial failures (*5*).

Pierre Poivre, the 18th century French explorer, in telling of his visit to Cochin China, about 1750, says (*6*):—

> "They cultivate the cotton plant— tea, indigo, saffron and, that which is theirs particularly, a plant which is called Tsai, which on being allowed to ferment as is done with that of

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mens de l'abondance et de la bonne agriculture; je ne l'ai vue florissante que dans les pays où ces deux droits de l'homme étaient bien établis. La terre qui multiplie ses dons avec une espèce de prodigalité sous des cultivateurs libres, semble se dessécher, même par la sueur des esclaves. Ainsi l'a voulu l'auteur de la nature qui a créé l'homme libre, et qui lui a abandonné la terre avec ordre que chacun cultivât sa possession à la sueur de son front, mais avec liberté.

Les cochinchinois suivent plusieurs autres cultures très-importantes, soit pour leurs fabriques intérieures, soit pour leur commerce au dehors.

Ils cultivent le cotonnier, le mûrier, le poivrier, l'arbre de vernis, l'arecquier, le thé, l'indigo, le *saffranum*, et, ce qui leur est particulier, une plante qu'ils nomment *tsai*, qui étant mise en fermentation comme celle de l'indigo, fournit abondamment une fleur de couleur verte, qui seule donne en teinture un verd d'émeraude très-solide.

Cette plante serait un présent bien essentiel à faire à nos colonies d'Amérique. Je serais trop long, si j'entreprenais de décrire

**Figure 1**
**Poivre's announcement of finding a green dyestuff in Cochin-China**

indigo, furnishes abundantly a flower of green color which alone gives in dyeing an emerald green very fast. This plant would be a present which might well be made to our colonies in America."

There are no records of anyone making use of Poivre's discovery.

In the search for a green dyestuff we might expect our old friend, Edward Bancroft, the dyer, spy, and discoverer of quercitron, to take a part. We find that we are right, for on going through his famous work on "Permanent Colours," we read on page 264 of Vol I, under the subject, Green Indigo (*7*):—

"About the year 1790, Mr Alderman Prinsep who had then lately returned from India, gave me a specimen of indigo obtained there, as he informed me, from a tree, and he, at the same time, gave me a very small piece of a hard green substance, likewise produced in the East Indies and which he called green indigo.

"Upon seeing it, I flattered myself with the hope of its proving to be what the late Mons De Poivre had mentioned in a little work published under the title of Voyages D'un Philosophie, as obtained by the inhabitants of Cochin China from a plant called Tsai, which, when macerated and fermented like indigo, yields a green fecula, capable of dying a fine, as well as a lasting emerald, or green colour. The quantity of this green substance so put into my hands, was much too small even for a single decisive experiment—But this is certain, that if a simple or homogeneous green coloring matter exists and can be discovered, with properties and other respects similar to those of indigo, it will be a most important addition to the Materia Tinctoria."

Bancroft further continues on the subject of green dyestuffs (*8*):—

"In the year 1793, Messrs John and Francis Baring, and Co received from R C Birch, Esq of Calcutta, parcels of two new drugs intended for dyeing; samples of which were put into my hands, with a request that I would make suitable trials of their merits; and with a paper containing some explanations which had accompanied them from India. One of these was called Barasat Verte and was formed into dry hard cakes, resembling in size and shape those of the indigo sent from Bengal; but of a dark dull green colour. It was stated to be a simple substance and to have been prepared with water and fire only, from an indigoferous plant, an evergreen, with leaves somewhat resembling those of the laurel. It was also represented as giving a durable light green colour, without any mordant or basis to silk and wool; and to be incapable of dying dark green without the aid of some blue colouring matter.

"After what has been just mentioned on the subject of green indigo, it will naturally be concluded, that my curiosity must have been greatly excited by that now under consideration; and indeed I lost not a single minute in making such a trial of it, as would decisively ascertain whether it really possessed the properties of indigo, with only the difference of a green instead of a blue colour."

After detailed experiments, Bancroft found that this so-called Barasat Verte was in effect simply an impure indigo, and concluded that it was (*9*):—

"Not likely to be of any use in dyeing; for though (the color) should prove lasting upon woollens, there are many other and much cheaper means already in use for giving colours of this kind to wool. It seems evident therefore, that the true nature of the supposed green indigo was but very little known to the discoverer thereof; and that its useless heterogeneous parts were the only ones which produced the colours dyed in Bengal, and which induced them to send it to Europe as a dyeing drug."

As a postscript to this project, Bancroft had this to say (*10*):—

"Mr Birch did not, however, relinquish this object until by sending to this country, several parcels of the Barasat Verte, and getting it used by dyers, he became fully satisfied, that

CHAMBRE DE COMMERCE DE LYON

NOTICE
SUR
VERT DE CHINE
ET DE LA TEINTURE EN VERT
CHEZ LES CHINOIS
PAR M. NATALIS RONDOT

SUIVIE
D'UNE ÉTUDE SUR LES PROPRIÉTÉS CHIMIQUES ET TINCTORIALES
DU LO-KAO
PAR M. J. PERSOZ

ET DE RECHERCHES SUR LA MATIÈRE COLORANTE DES DIFFÉRENTS INDIGOÏDES
PAR M. A.-F. MICHEL

IMPRIMÉ PAR ORDRE DE LA CHAMBRE

PARIS
TYPOGRAPHIE DE CH. LAHURE ET Cie
IMPRIMEURS DU SÉNAT ET DE LA COUR DE CASSATION
RUE DE VAUGIRARD, 9
1858

**Figure 2**
**Title page of work on Lo-Kao published by Chamber of Commerce of Lyon in 1858**

it had no other value than that of the indigo contained in it; and that, as indigo, this was not the most advantageous form, or method of preparing it."

Almost 50 years more had to pass until we come to our real story—the discovery by the Western World of a green dyestuff, Lo-Kao, in China, in the middle of the last century.

There are many facets to this discovery and to its development commercially in Europe. Perhaps the story is best begun with a document as historically important in its own way as Perkin's original patent on Mauve. I refer to the paper presented by Persoz, the famous dye chemist, before the French Academy of Sciences on the 18th of October, 1852 (*11*). The English translation follows:—

"I have the honor to deposit on the desk of the Academy a specimen of a coloring matter used in China to dye textile fibers green. The Academy will permit me to review in a few words how I came to ascertain the existence of this color.

"Mr Daniel Koechlin-Schouch, having sent me last autumn a sample of calico dyed in China a green of high stability, asked me to study the composition of this green color. All of the tests which I made on this sample in an attempt to determine either blue or yellow resulted in failure and I was convinced on isolating the coloring principle that this green was due to a dyeing material of one par-

**Figure 3**
**Rhamnus Utilis—Source of Lo-Kao**

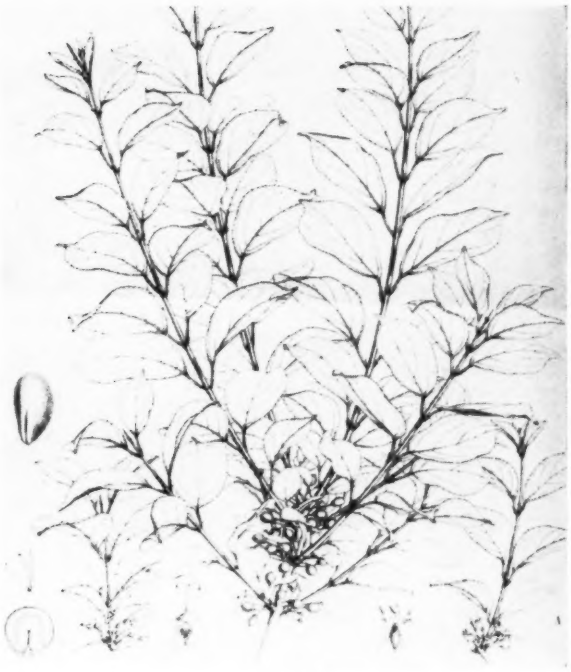

**Figure 4**
**Rhamnus Chlorophorus—Source of Lo-Kao**

ticular nature and of one particular composition. It became evident:—

1. That this coloring material was of an organic and vegetable origin.

2. That the fabric on which it was fixed was found to contain a larger amount of aluminum and a little oxide of iron and of calcium, substances whose presence indicate necessarily that in order to adhere to the fabric, the coloring material used must have needed mordants.

"These results so positive and at the same time so different not only from what we have known in Europe concerning the composition of greens, but also different from all which has been written on the dyeing processes in use among the Chinese to make this color, necessitated on my part a much deeper examination; accordingly towards the end of the month of November last, having recourse to the kindness of Mr Forbes, American Consul at Canton, I requested a specimen of the precious material. He had the goodness of sending me about a gram.

"This substance consisted of thin plates of a blue color being very similar to the indigo of Java, but it consisted of a much finer paste and was quite different from indigo both in its composition and in all its chemical properties. After having infused a very small piece of this substance in water, this medium did not delay in becoming colored dark blue with a greenish reflection. The liquor brought progressively to the boil accomplished a true dyeing on putting into it a sample of calico on which had been printed mordants of iron and aluminum and one noted that—

The parts of the fabric containing aluminum became green more or less heavy depending on the intensity of the mordant;

The parts covered with aluminum and iron oxide became a strong green of olive shade;

The parts finally containing pure iron oxide only became a strong violet;

The parts of the fabric not containing any mordant remained essentially white.

The colors thus obtained were treated with all of the agents which we had previously used on the Chinese green, and the results proved the same. From these experiments one concludes;

First, the Chinese possess a dyestuff having the physical aspects of indigo which colors green the mordants aluminum and iron.

Second, that this coloring material does not contain indigo nor any derivative of this dyeing principal.

"The honorable president of the Chamber of Commerce of Paris, Monsieur Legentil, having so much interest for science and industry and so that our country might promptly come in possesssion of this precious material, several months ago took all the necessary measures in order to obtain as quickly as possible a certain quantity and to obtain at the same time all information concerning its origin and its preparation.

"I expect to submit to the academy a complete work on this new color after I have made a more detailed study."

After Persoz' announcement, things began to move quickly. Here at last was the green dyestuff—sought for hundreds of years. The difficulties of importation from China could not stand in the way. The dyers were clamoring for this "wonder" dyestuff. Cost was no object. By 1853 quantities of this china green, known as Lo-Kao, were coming to Holland and to England as well as to France. In England, John Mercer and Walter Krum began detailed investigations of the color. In France, other eminent chemists besides Persoz became enamored with Lo-Kao and started their special investigations (*12*).

As one would expect, even after Lo-Kao became an important article of export from China, its actual preparation as carried on in China was surrounded in mystery. This is expected when one realized how difficult it was to get any technical information out of the Far East in the middle of the last century. Westerners were not permitted, generally, to travel into the interior and the language difficulties alone served as a great barrier. Nevertheless, some of the missionaries and commercial rep-

**Figure 5**
**Production of Lo-Kao—Steeping of fabric**
**(Rice paper painting)**

**Figure 6**
**Production of Lo-Kao—Exposing fabric to the sun**
**(Rice paper painting)**

resentatives were able to obtain a reasonable story as to the origin and method of preparation of Lo-Kao.

Apparently, two different plants were needed to prepare the Lo-Kao of commerce. The two plants were *Rhamnus Utilis* and *Rhamnus Chlorophorus*. One of these shrubs produced a deep and fast color without too much brilliancy while the other furnished a weak but very brilliant color. The combination of these two gave the valuable green color of Lo-Kao.

As always seems to be true of technical operations in China, the methods for the preparation of the dyestuff were laborious and round-about. The dyestuff was actually obtained by first dyeing cotton cloth in a solution containing the color extracted from the two plants and then extracting this excess color from the dyed cloth.

The missionary and explorer Father Helot said that he witnessed the following process at A-Ze (*13*):—

> A color extract was made from each of the two plants. The infusions actually required quite a few days to prepare. Cotton cloth was dipped seven to ten times in one bath with a drying after each immersion. Then this cloth would be dipped in the other bath three times and again dried after each immersion. The drying took place by spreading the dyed cottons out in the fields at nightfall and leaving them until noon the next day The action of the sun was necessary in the development of the color. Now, after the cloth had been dyed as above by this long and laborious process, it was then placed in cold water and thoroughly rinsed. The wash waters were collected together and then boiled in a large cauldron. Over the top of the cauldron was spread a network of cotton yarn upon which the coloring matter becomes deposited during the boiling process. In order to collect a reasonable quantity of the dyestuff this process of boiling had to be continually repeated with fresh quantities of water in which the previously dyed cloth had been washed. When enough insoluble color had collected on the cotton yarn, the latter was then washed in cold water and rubbed with the hands by which treatment the Lo-Kao breaks away and slowly settles down in the water. This insoluble dye was washed by decantation and the thin pasty mass placed upon paper and dried. The thin layer of Lo-Kao was detached after drying and this became the small platelets as were met with in commerce.

It is said that the weight of cotton yarn to exhaust the pigment from 300 pieces of dyed cloth was 3.62 kilograms. In order to obtain 1 kilogram of Lo-Kao, 1,060 pieces of dyed cloth were required, and, according to Helot, the five factories for Lo-Kao at A-Ze could only yield a total of from 18 to 24 kilograms per year. It is readily understood why Lo-Kao was so high priced in spite of the low cost of labor in China (*13, 14*).

Despite the high price and despite the difficult procedures used in dyeing silk with Lo-Kao, for some ten years after Persoz' announcement, this dyestuff was imported in considerable quantities and widely used. Even the possibility of cultivating European *Rhamnus* plants was given consideration. The days of Lo-Kao were numbered, however. Perkin's discovery of Mauve marked the beginning of the end of the natural dyestuffs.

The first to go was not indigo nor weld nor cochineal but Lo-Kao. The latest on the scene was the earliest to leave, for by 1860 the first aniline green had been discovered. Within a short time aldehyde green and then iodine green were on the market. (*15*). After that, Lo-Kao never had a chance. It quickly disappeared into obscurity only to be thought of today as an exciting story in the history of dyeing.

## REFERENCES


(1) "The Whole Art of Dying," London (1705).
(2) "The Art of Dyeing Wool, Silk, and Cotton." Translated from the French of M Hellot, M Macquer and M Le Pileur D'Apligny; London (1789).
(3) Berthollet, "Elements of the Art of Dyeing." Vol II, p 308. Translated from the French by William Hamilton; London (1791).
(4) "A Practical Treatise on Dyeing and Calico-Printing," p 358; New York (1946).
(5) Chambre De Commerce De Lyon, "Notice Du Vert De Chine." p 20; Paris (1858).
(6) "Oeuvres Complettes de P Poivre," p 159; Paris (1797).
(7) Bancroft, Edward, "Experimental Researches Concerning the Philosophy of Permanent Colours," Vol I, p 264; London (1813).
(8) Ibid, p 266.
(9) Ibid, p 271.
(10) Ibid, p 274.
(11) Persoz, J, "Sur Une Matiere Colorante Verte," *Comptes Rendus* **XXXV**, 558-559; Paris (1852).
(12) Chambre De Commerce De Lyon, "Notice Du Vert De Chine."
(13) Crookes, William, "A Practical Handbook of Dyeing and Calico Printing." p 428-434; London (1874).
(14) Calvert, F Crace, "Improvements and Progress in Dyeing and Calico Printing since 1851"; Manchester (1861).
(15) Reimann, "Aniline and its Derivatives"; New York (1868).

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# NONWOVEN FABRICS
## The Chemical and Processing Techniques*

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### INTRODUCTION

THE recent growth and expanding interest in the manufacture of nonwoven fabrics represents one of the most significant departures in the textile industry in a number of years. Textile and chemical technology have been combined in order to produce a wide variety of versatile end products suitable for an unlimited number of industrial, domestic and personal uses. Nonwoven fabrics are by no means a new commodity; however, newer techniques and processes have enabled manufacturers to produce a more complete line of fabrics and have allowed definition of many new end uses. The original development of nonwoven fabrics has been discussed frequently and need not be further mentioned herein. At this time an attempt will be made to present a comprehensive set of data on the manufacture, end use, and potential of many nonwoven fabrics of specific types.

### DISCUSSION

FIBERS———During the past year, sixty to sixty-five million pounds of cellulosic and synthetic fibers have been utilized in the manufacture of nonwoven fabrics. Of this volume, cotton and rayon constitute 90% to 95% of the total. The newer synthetic fibers, however, are gaining more prominence in this field and offer properties not entirely obtainable with cellulosics. Nylon and acetate are the two most frequently encountered synthetics. The addition of nylon to the web significantly enhances the general physical strength of the bonded fabric. The necessity for synthetic fibers will become more prominent with the growing interest for the use of bonded fabrics as interlinings for wash-wear suitings. A rapid drying rate will be provided with a minimum of dimensional distortion. Many of the present interlining fabrics will contain from 20% to 50% of the polyamide fiber with current attention being directed towards substantial increases in tensile and tear strength. A great deal of interest is presently being directed toward the formation of bonded fabrics capable of being tufted in order to replace the woven jute fabrics commonly used as rug backings. Here too, the incorporation of nylon is of significant importance in that in the tufting operation high strength, as well as mobility of the fiber, is important.

Acetate is also gaining prominence in this field. Although the strength imparted does not approach that of nylon, secondary acetate does provide functionality to the fabric with regard to both hand and appearance. Acetate, always considered a fashion fiber, should find expanded uses in this field.

In view of cost, certain limitations have been placed on acrylic and polyester fibers. The latter, however, exhibit properties not obtainable with other synthetic fibers when factors such as acid-, alkali- and oil-resistance, as well as stability and electrical properties, are necessary. The polyester fiber makes definite contributions to industrial purposes such as filters, gaskets, etc.

FORMATION OF THE WEB——— Synthetic and cellulosic fibers are mechanically processed in one of three principal methods in order to form bonded fabrics. As a general rule, the fiber processed will have a staple length of approximately ¾" to 1½". Following opening and picking, the webs are presently formed by the card, garnett or Rando-Webber (*1*). In many cases it is difficult to describe why one unit has an advantage over the other due to the fact that versatility with existing equipment and imagination of the operator play an important role. In general, for quality fabrics, a card is used rather than a garnett. The rate of productivity is not high; however, uniform fiber lay with little variation is obtained. For quality fabrics of one to two ounces per square yard, a capacity of 25 to 45 lbs per hour may be expected. The capacity may be increased substantially when weight per square yard is increased, while sacrificing appearance and uniformity. The garnett, while considered inferior to the card for these lighter webs, is capable of high speed, and for battings, packings, etc, as much as 300 to 400 lbs may be produced in a single hour. At a lower rate of productivity, however, a garnett will not provide strength or uniformity in the unbonded web comparable to that of the carded web for the before-mentioned high-quality, lightweight webs.

Either carded or garnetted webs are normally cross-lapped when strength is desired in all directions. This is essentially a distribution of the strength obtained in the machine direction so that a substantial increase in the unbonded web and bonded web is obtained across the fabric. In the unbonded state the average strength in any direction after crosslapping is usually less than that obtained in the machine direction of the web which has not been crosslapped. Surprisingly good uniformity and tensile strength is obtained with a bonded cellulosic or cellulosic-synthetic web which has been carded and handled in this fashion.

The Rando-Webber (*1*) represents a relatively new type of web former. This unit produces web with equal

* Presented before the Mid-West Section at the Netherland Hilton Hotel, Cincinnati, O, on April 27, 1957.

strength in all directions without crosslapping. The Rando-Webber will process stock at a rate of up to 300 lbs per hour on a 40" machine. Here again, we are concerned with the matter of quality in that a relatively high-strength, uniform web, in the one- to two-ounce category, would be produced at a rate of 40 to 70 lbs per hour. The Rando-Webber is capable of processing a large variety of fibers, as well as processing wastes. In the latter cases uniform webs have been produced from garnetted threads, leather, paper shoddy, foam, yarn wastes, etc. A greater degree of versatility is offered with this unit and the prehandling can be kept at a minimum.

Still a newer type (2) being proposed for the manufacture of bonded fabrics is a modified card utilizing an air-doffer principle. This unit involves feeding the open stock into a single card, or a duplex unit of two cards, feeding into a second of greater width, and then air doffing to avoid distortion and obtain uniformity of the finished web.

It is difficult to prescribe any specific unit for this industry. Unquestionably, quality or quantity may be obtained on each of the above systems. This will depend primarily on the type of fabric, or fabrics, to be manufactured with regards to economy, availability and uniformity.

APPLICATION OF RESIN——— Following the formation and fabrication of the web, synthetic polymeric emulsions are applied by either impregnation, foaming, spraying, printing or kiss roll. At the present time there are only two saturators available to the industry on a commercial scale. Due to the fact that the fibers are in a fluid state immediately after saturation and immediately during nip-roll compression, some protection is necessary in order to avoid accentuated distortion of the mat. A saturator has been devised (3) in which the web is saturated between two screens and the excess liquor removed by a set of nip rolls. The web is still contained within the screen during the compression and up to a point where adequate strength is provided by the bonding agent.

Still another method (4) involves saturation of the web by passing it around a perforated cylinder which is immersed in the emulsion. The web in this case is supported by a single screen and held intact with the surface of the cylinder. Following the saturation, the excess liquor is removed by vacuum extraction. During the extraction cycle, greater penetration of the resin into the stock is assured because the liquor is transferred directly through the web.

Several years ago a patented process (5) was presented which involved foaming of an emulsion polymer onto a web. This procedure was described in the patents as gravity feeding of a foam on the surface of the fabric, the excess being removed by a doctor blade. Such a method is claimed to allow uniform application of resin with a minimum degree of web distortion. Resin may be applied in succeeding operations until the desired strength is obtained to enable handling the goods as a woven fabric. Polymer may also be applied to the web by a print roller which provides localized bonding. Such techniques would be used primarily for disposable items since adequate strength for repeated processing is not provided. Fabrics prepared by the above principles afford an item relatively compact and approaching the behavior of woven fabrics. There are cases where resistance to laundering and drycleaning are not essential and where loftiness is a desirable characteristic. In these particular cases a degree of original tensile strength is desirable, and rather than impregnate the fabric and lose this loft, the web either may be sprayed or coated on the surface by a kiss roll. In the former case, webs may range from ½" to 2" in thickness. Common techniques would involve spraying an extremely fine mist of emulsion onto one side of the fabric, drying, then reversing the fabric and spraying the other side, followed by a second drying operation. Webs produced in this fashion have relatively good tensile strength, uniformity of resin and resilience. In fabrics of this type, end uses may call for deposition of the resin on the surface for applications such as dielectric heat sealing.

An additional technique for imparting increased tensile strength is to pass the fabric over a kiss roll which revolves in the treating liquor. The use of the product may demand application of the bonding material to either one side or two. Since fabrics of this type are not subjected to extreme conditions, the lack of bonding agent in the center of the web is not important.

When webs are saturated by either of the two above-mentioned methods, consideration must be given to the desired hand of the finished fabric. Where smoothness of hand or a greater surface effect is desired, application of the resin can be made in two or more steps. In some cases it may be desirable to avoid applying all of the resin in one pass since varied hand effects may be required which may involve a two- or three-stage application. This perhaps could be avoided in many cases if the degree of wet pick-up would be similar to that involved for a woven fabric. However, in some cases wet pick-up may range from 300% to 600% and deposition of the polymer under conditions such as this will cause undesirable properties for specific fabrics. Versatility in hand can be accomplished by both hot-air and hot-can drying.

It should be mentioned that the techniques described above for formation of the web, as well as the application of resin, are representative of the more commonly known and frequently discussed methods of manufacture. Without question, there have been departures from these techniques which offer specific advantages in the finished product. Many of these modifications, whether minor or major, are not disclosed since they are representative of achievements at the manufacturing level which offer the producer significant advantages in production.

DRYING———Much consideration must be given to the drying techniques involved with the above methods of application of resin. Migration of the polymer on webs which have been saturated must be carefully controlled. If the webs contain a high degree of polymer and water, the same precautions should be followed in the initial stages of drying as those for woven fabrics. The escape of water encourages shifting of the polymeric particles to the heated surfaces of the fabric thus causing a "hungry" area. An ideal case would then be that of an emulsion which is relatively stable for processing conditions but which will coagulate rapidly when deposited on the fabric. In air drying, the early stages should be confined to a temperature of 200 to 260°F. Roughly 50% of the water should be allowed to evaporate before the web is exposed to temperatures in excess of 300° F. Although many thermoplastic polymers are adequately fixed with drying alone, exposure to a high temperature in the vicinity of 300° F is favorable in order to allow greater flow and subsequent bonding of the

staple fiber. In cases, however, where thermoplastic polymers are used in conjunction with thermosetting resins, a temperature in excess of 280° F is necessary in order to allow the thermosetting resin to crosslink with itself, the fiber, and, in some cases, the polymer. Can drying is also an entirely favorable operation, but, here, too, consideration must be given toward migratory properties of the resin. It is advisable to maintain the temperature of the first two or three cans below 250° F and then heat the remaining portion of the battery to a maximum practical temperature. When drying on cans, deposition of the polymer can usually be avoided by the use of either Teflon or silicone coatings. Since the fabric contains such high concentrations of polymer, one might anticipate a self-cleansing action during the early stages of drying.

BONDING AGENTS —— Since, in many cases, the bonding agent employed constitutes a major portion of the weight of the fabric, many factors must be considered in the selection of the proper resin. For usage in apparel, for example, concentrations of 40% to 50% resin on the finished weight of the fabric are common. There is a considerable number of specific types of polymers, and each classification thereof offers inherent properties on which subsequent fabrics are dependent. Both thermoplastic and thermosetting resins play an important role, and combinations thereof deserve critical attention.

Properties such as mechanical stability, particle size, flexural rigidity, stability to ultra-violet light and heat, migratory behavior, heat sealability, adhesion and economy must be given due consideration. When a certain chemical type is being considered, however, it must not be construed that all emulsions within that type have similar behavior. As an example, polyvinyl chloride polymers may present aging properties ranging from poor to good, nitrile latices provide poor-to-fair solvent resistance, and acrylic polymers poor-to-excellent washfastness. In essence, each polymer must be handled as an entity since differences exist within a rubber group as well as between two different groups.

Synthetic polymers generally used for the manufacture of nonwoven fabrics are susceptible to various chemical and physical changes if care is not taken in the manufacture of the emulsion or in its use. For current methods of high-speed production and the versatility demanded of the resins, mechanical stability of the emulsion is an important factor. Resistance of the polymers to coagulation during processing is primarily a function of the soap system used for emulsification, particle size, uniformity and pH. It is no accident that the predominent number of synthetic polymeric emulsions are supplied on the alkaline side. By nature, the emulsion stability is markedly improved by the addition of various bases in order to obtain a pH of 7 or higher. No generalities can be formed regarding the type of soap system used to emulsify an unlimited number of monomers. A sulfonated ethylene oxide condensate may be an ineffective emulsifying agent for methylacrylate but entirely satisfactory for butadiene-acrylonitrile polymer. In the manufacture of the emulsion the emulsifying agent plays a role capable of causing numerous side effects. The rate of reactivity, molecular weight, average distribution of particle size, etc, are factors constantly plaguing the emulsion chemist. Each polymer is handled as an entity and systems are employed which provide optimum properties for the intended use of the polymer.

Some synthetic polymeric emulsions are attacked and subsequently breed bacteria when used under adverse conditions. Additions of bactericide, alkali or acid will readily control this growth. The acid addition, however, as mentioned earlier, will be dependent on the possible detriments it may cause the emulsion. There is no doubt regarding the occurrence of bacteria; obnoxious odor accompanied by a blue color is obvious. This blue color, incidentally, is markedly different in intensity from the bluish optical cast caused by the preparation of emulsions with infinitely small and uniform particles. Formaldehyde, as well as the above-mentioned agents, may be employed to avoid the initial attack of bacteria, or as a later addition to avoid further growth. Additions of formaldehyde to a contaminated emulsion will retard further growth but will not offset the chemical degradation caused by bacterial action.

Oxidation of the polymer and accompanying physical changes of the nonwoven fabric is a factor deserving utmost attention. Natural rubber and many synthetic rubbers require antioxidents to minimize the occurrence of brittleness, discoloration, etc. These unsaturated linear polymers are readily attacked by small quantities of oxidizing agents. From a textile point of view this property is a severe detriment and limits the potential use of many synthetic polymers. In contrast, selectively stabilized polyvinyl chloride emulsions show good behavior in this respect and the fully saturated acrylic polymers or copolymers are optimum with regard to resistance to oxidation processes.

The degree of firmness or softness imparted to a textile fabric is obtained in many ways. Specifically, for nonwoven fabrics, drapeability is obtained with polymers which have been plasticized either internally or externally. The former, of course, is more desirable since plasticization has been accomplished by building the softer monomer (acting as plasticizer) into the resultant polymer in any desired ratio. Externally plasticized polymers, on the other hand, require caution in use. Due to the lack of homogeniety, migration of the plasticizer is quite common. An externally plasticized polymer has restrictions on a web destined for apparel since loss of the plasticizer on laundering or with chlorinated solvents will cause a marked increase in the rigidity of the web.

TYPES OF BONDING AGENTS ——Of the synthetic polymers encountered, the following represent those used in greatest volume for nonwoven fabric manufacture: polyvinyl chloride, acrylic, butadiene-acrylonitrile or nitrile latices, butadiene styrene, and polyvinyl acetate.

Polymers of vinyl chloride show marked color changes and increasing stiffness unless suitable stabilizers are used. This discoloration is caused by the loss of the halogen and formation of hydrochloric acid, which, combined with oxygen, attacks the newly formed double bonds. The stabilizers utilized either accept the liberated acid and or saturate the recently formed double bond. As a class the polyvinyl chloride polymers must be characterized as being not resistant to oxidation; therefore, marked discoloration and stiffness occurs. Recent work, however, has led to the development of newer and improved types which are internally plasticized and relatively resistant to degradation by ultra-violet light and heat. The introduction of these features has broadened the potential use of these polymers as bonding

agents for nonwoven fabrics. The polyvinyl chloride polymers provide a low degree of elongation with fairly good tensile strength. Drycleanability of the group will range from fair to good, although washfastness in many cases is marginal. In general, the newer types of polyvinyl chloride polymers have made contributions to the bonded fiber industry. Economy and general properties in use have substantially increased the market of these polymers.

The acrylic polymers, copolymers, terpolymers, etc, have shown continued growth in the bonded fiber industry. Until recent years these materials were characterized by good performance, but they were considered excessive in cost. Technological improvements in the manufacture of the monomers and subsequent polymers has offset the pre-existing price differential. In most cases they are now competitive with the conventional latices employed in this industry and provide inherent properties desirable in the manufacture of nonwoven fabrics. The acrylic polymers are fully saturated and degradation of the polymer in normal use is nonexistent. Since they are fully polymerized, and thereby saturated linear polymers, oxidation processes do not affect the appearance or stiffness of the film. The acrylic polymers are internally plasticized and any degree of flexibility may be obtained. As in the conventional emulsion polymerization, the ratio of monomer is the determining factor in providing soft or firm film characteristics. The new type acrylic polymers offer optimum conditions regarding mechanical stability. Of this newer group, the polymers provide good durability to laundering and drycleaning. As may be expected, this durability is significantly enhanced by the use of thermosetting resins to provide a crosslinking mechanism. The versatility of the acrylic polymers is apparent since their general properties allow them to be used in all the conventional processes for the preparation of nonwoven fabrics. Among the softer varieties, good tensile strength is obtained, with fair-to-good tear strength imparted to cellulosic and synthetic webs. Of the softer variety, good resilience is obtainable; however, the rate of recovery is somewhat slower than that obtained with several of the synthetic rubbers. Although the rate is somewhat slower, the degree of recovery is equally as great, thus allowing treatment of the web as a wash-wear fabric. It is anticipated that the newer types will have the characteristic bouncy feel while still maintaining the properties required to obtain the intrinsic behavior of a textile fabric.

Of the types mentioned above, reference is made to acrylic polymers which are solely based on acrylic monomers, such as methylacrylate, ethylacrylate, methylmethacrylate, etc. In addition to this type, the term *acrylic ester* has become common in a different sense of the word. Although the term *acrylic esters* would be definitive of the above type, it could also include copolymers of an acrylic monomer and a foreign synthetic latice to perhaps provide intermediate effects. Acrylic monomers may be copolymerized with numerous synthetic monomers to meet specific end-use requirements. Such products have found wide use; however, their over-all properties do not quite match the behavior of the acrylic polymers *per se*.

Butadiene - acrylonitrile latices, when used as bonding agents for nonwoven fabrics, provide good-to-excellent tear strength on webs composed of both cellulosic and synthetic fibers. This degree of tear strength is accompanied by fair tensile strength and softness of hand where desirable. The nitrile latices as a group are susceptible to oxidation. Here again, within a chemical class, degradation of the acrylonitrile latices, when exposed to ultra-violet light or heat, ranges from extreme-to-moderate change in color and/or stiffness. Without question, this property somewhat restricts the use of these polymers from select applications where stability is necessary. Aside from this, in fields where such aging properties are not essential, the nitrile latices are used extensively where economy, mechanical stability, elongational behavior and versatility are attractive. The launderability of these polymers may range from fair to good, and in general the drycleanfastness is rated as equally good. With these materials, the addition of thermosetting resins also enhances these latter properties to a marked degree.

Polymers based on butadiene-styrene offer a factor of economy which cannot be overlooked in this industry. In general use, the restrictions are obvious due to the relatively poor mechanical stability of many in this class and the residual tack, which all too frequently occurs. Butadiene-styrene polymers are the most rapidly attacked by heat and ultra-violet, and severe discoloration, with a substantial increase in firmness, is noted. Further work will undoubtedly lead to improved types in which some of these factors may be offset. Tremendous quantities of these polymers are consumed in unrelated applications in the textile industry, and although there may not be a direct correlation to nonwovens, improvements may be anticipated which might reduce the limitations in this field.

Polyvinyl acetate has been a conventional finishing agent in the woven fabric industry for many years. It shows promise in select fields in the manufacture of nonwoven fabrics also, and its general behavior in application and use is attractive for these outlets. Webs bonded with polyvinyl acetate lack the resilience which, in many cases, is desirable in this field. The degree of recovery is relatively poor and the adhesion to many of the synthetic fibers is marginal. Fastness to laundering and drycleaning ranges from poor to fair.

The comments pertaining to the above synthetic latices are primarily in reference to their use as a self-bonding agent. In most cases the fastness properties are substantially improved by the addition of thermosetting resins of either the ethylene urea - formaldehyde, melamine - formaldehyde, methylated urea-formaldehyde or epoxy type resins. For example, certain of the acrylic polymers contain functional groups capable of crosslinking with themselves or with the thermosetting additive. Combination of the thermoplast and thermoset thereby minimizes swellability in solvents, further reduces any residual tack, and adds a greater degree of resilience to the fabric. Where a minimum change of hand is desired, the ethylene urea derivatives are suggested due to their inability to react with themselves and the fact that crosslinking occurs only with the thermoplastic polymer or the fiber. The use of thermosetting resins of this type will require further consideration in the curing of the finished fabric. In most cases temperatures of 300° F or higher are desirable in order to obtain a higher yield of reactivity between resins. Conventional catalysis is suggested, and metallic salts, ammonium salts, as well as amine hydrochloride catalysts are adequate. The reactive resin, depending on its chemical behavior, will be used either in the original bonding of the fiber in conjunction with the thermoplast, or as

a subsequent top treatment. Several factors must be considered in the selection of the thermosetting resin with each specific synthetic latice. These factors are: flexibility, resistanee to chlorination for special end uses, economy, discoloration, and rate of reactivity. As mentioned above, the effect of thermosetting resins used in conjunction with latices is most apparent when the latter agents exhibit some functionality and ability to crosslink either with themselves or with the thermosetting resin. In general, the ratio of thermoplast to thermoset resin will range from 8.5/1.5 to 9.5/.5. A compromise is sometimes essential in order to impart minimum hand with certain of the thermosetting resins while improving the durability of the polymer to the desired degree. For example, in a web composed of nylon and acetate, the melamine-formaldehyde resins as well as the methylated urea-formaldehyde condensates tend to give a degree of crispness and firmness to the fiber.

In many cases, nonwoven fabrics will be exposed to chlorination procedures similiar to those encountered for woven goods. In essence, the behavior of the thermosetting resin on nonwovens parallels its property on woven fabrics. In this respect the epoxy resins, because of their nonnitrogenous nature, exhibit no chlorine degradation.

Both the melamine and ethylene urea derivatives will pick up and retain chlorine; however, with hot chlorination, discoloration is markedly less with the latter material. On some nonwoven fabrics, because of the location of the thermosetting resin, discoloration may be more noticeable than on woven goods. This may be due to the fact that prebonding techniques with elastomer alone restrict the thermoset from the interstices of the fiber and a greater portion is deposited on the surface where it is more rapidly attacked by chlorine. Although discoloration may occur, it is not associated with high strength loss as in the case of woven fabrics. While the same mechanism occurs in the release of hydrochloric acid, the high concentrations of thermoplast tend to offset the actual degradation of the cellulosic fibers. The conventional urea-formaldehyde paste may be used satisfactorily; however, the limited storage stability and greater potential of odor development on the cured fabric are restricting factors.

Further consideration is being given to thermosetting and thermoplastic resins used in other industries as these may be readily adaptable to the subject application. For example, many manufacturers desire bonding agents capable of resistance to chemical and physical degradation comparable to the fiber which they are bonding. For numerous industrial applications, bonding agents resistant to acid, alkali, oils, etc, would allow greater versatility for the production of industrial filters, gaskets, etc. Some attention has been given to the use of polyester-styrene resins, which may provide chemical resistance comparable to Dacron polyester fiber (6).

Further, nonwoven fabrics which are subsequently used to prepare laminates would be more receptive to lamination techniques if the bonding agent for the fiber was chemically similar or identical to the laminating resin. Since styrene-polyester resins are normally used for this purpose, special steps may be required for emulsification, application, etc. The styrene is not a diluent for these resins but a coreactant for the polyester. Factors such as air inhibition should not be as critical in handling of the web as for molding or lamination of the same resin.

The monomeric and polymeric plasticizers enhance the heat sealability factors of numerous thermoplastic latices. Plasticizers will change the power factor of the film, thus allowing a greater degree of excitement of the polymer under standardized heat-sealing frequencies. Dibutyl phthalate and dioctyl sebacate are two monomeric types which exhibit this property. Alkyd resin may be utilized to provide improved fastness properties to the polymer and, perhaps, to the selective coloring agents which may be used in the saturation. The incorporation of pigments and/or direct dyestuffs to the saturating system provides a method to uniformly dye the fabrics as well as to apply required amounts of bonding agent. Since, in most cases, the ratio of thermoplast to pigment is extremely high, further protection may not be necessary depending upon the use for which the fabric is destined. Chemical suppliers are constantly striving to offer new and improved types of resins for this industry, as is evidenced from past work. A great deal of the limitations of the fabric are primarily functions of the bonding agents employed. The combined efforts of supplier and consumer has led to definite advancements in this direction.

BATTINGS, WADDINGS, PACKINGS, ETC———For a number of years thick webs composed primarily of cellulosic, but in some cases synthetic, fibers have been produced for various industrial, domestic and apparel uses. Webs of this type may vary in thickness from approximately one-half inch to two inches. These webs, as described earlier, generally are produced on a garnett at speeds up to 300 lbs per hour. Depending on specific end uses, they may be used directly from the garnett or after subsequent wet- or dry-bonding techniques. The end use primarily will dictate the finishing techniques required. In many cases adequate strength is imparted to webs of this type by processing on a needle loom. This method involves entanglement of the fiber at multiple locations to increase the tensile strength in order to obtain satisfactory processing conditions. Since the loftiness must be maintained, the tendency is toward dry-powder bonding in order to obtain the desired properties. These products may be based either on thermoplastic or thermosetting systems. The procedure for distribution of the bonding agent is also dependent on the degree of uniformity required and the end use.

One such patented method (7) involves the sprinkling of powder on a light garnetted web immediately prior to crosslaping. Such a technique allows distribution of the resin between each individual layer and insures greater bonding efficiency and economy. After crosslapping, the thickness is obtained by varying the speed of the crosslapping belt or screen. The web is then carried into the heated chamber, and on slight compression fusion of the polymers occurs, thus substantially increasing the strength of the web. Thermoplastic powders are the principal chemicals used in this operation. Copolymers of vinyl chloride and vinyl acetate may be used effectively.

A newer type, based solely on acrylic monomers, has been developed and may offer intrinsic value to webs bonded with these materials. The latter are supplied in bead form and have a relatively uniform particle size. Discoloration of the polymer is avoided, thus allowing processing at substantially higher and more efficient temperatures. Of significant importance is the fact that it is possible to vary the average particle size of the polymers in order to provide versatility in related applications. It can be seen microscopically that fu-

sion of the particles occurs at the junction of the fiber and a degree of flow may be noted along the length of the individual fibers. Webs bonded by these techniques generally contain from 15% to 30% thermoplastic powder or bead based on the final weight of the fabric. It is conceivable that compressed webs with greater drapeability could be obtained by similar techniques since film continuity is avoided.

Phenol formaldehyde condensates may also be used in such a process. They have application only where discoloration of the web is not a hindrance since the conventional phenol trait is quite obvious. The economy, however, is attractive for many industrial applications, and for these uses these materials aptly provide satisfactory behavior.

Lofty webs commonly used in the quilting trade frequently require the addition of bonding agent in order to provide sufficient strength for the quilting operation. Loft is essential in order to maintain the intrinsic behavior of the web for apparel usage. There is evidence that, due to the physical form of the acrylic bead polymers, they may be uniformly applied to a thick web by gravity feed, provided it is not excessively dense. The filtration of the bead occurs throughout the web, thus allowing localized bonding sites.

The techniques described above are fairly well established; however, the increasing availability of new type bonding agents in solid form may open up new avenues for the manufacture and distribution of these items.

FIBER BONDING——A great deal of attention is being directed toward the development of improved synthetic thermoplastic fibers, which may be used effectively as the bonding agent for similar as well as different fibrous materials. Such techniques would allow the preparation of a completely fibrous nonwoven fabric, the thermoplastic fiber being used at varying concentrations on the total weight of the web in order to impart the desired degree of strength, drapeability, appearance, etc. Webs of this type would be prepared in the normal fashion after the fiber in question is mixed uniformly. After the web is formed, it may then be compressed during a heating cycle at a temperature in excess of the fusion point of the thermoplastic fiber. This then allows bonding at the crossover points of the thermoplastic fiber and the fiber to be bonded. It is expected that minimum hand buildup will be obtained by proper fiber selectivity and that the integrity of a textile fabric will be more obvious. Such procedures are common presently in the felt industry, and without question an extremely high degree of strength is obtained; however, more is to be desired with regard to softness and drapeability. It is quite likely that, in the near future, thermoplastic fibers will make more definite inroads into the bonded fiber industry and presumably open up newer and perhaps more difficult markets.

PAPER PROCESS——There has appeared recently on the market bonded fabrics manufactured by paper processes. Such techniques offer the possibility of producing bonded fabrics at a fantastically higher rate of speed than is normally encountered in the textile industry. A great deal of attention is being directed to this effort in certain quarters and the results to date have been quite promising for specific outlets. Both fiber and latex manufacturers have considered many specific means of bonding fibers for this industry. Beater additions, as well as impregnations or sprayings after formation of the web, etc, all have been thoroughly exploited. The major types available thus far still have the inherent properties of a papery item and the softness and drapeability normally associated with textile fabrics has not been obtained, or at least produced commercially. Closely related to textile and paper applications is the formation of papers composed of synthetic fibers. Short staple is processed for this use, usually in the range of ¼″ to ⅜″ in length. The fiber is dispersed, and after formation of the substrate, latices are applied. There is a great deal of related technology involved in both the paper and textile techniques and unquestionably there shortly will be some overlapping of the two independent operations.

## PRESENT AND FUTURE

A recent publication (8) has attempted to summarize the actual consumption of nonwoven fabrics during the 1956 era. The markets mentioned were as follows:

| | Million lbs |
|---|---|
| Paddings and packings | 20 |
| Industrial wipers | 1.0 |
| Coated fabrics | 25.0 |
| Disposable items | 12.0 |
| Apparel usage | 7.5 |

At the present time, the greatest growth potential appears to be in the ourwear field. Since little progress has been made thus far in producing items of this type, it is generally agreed upon that bonded fabrics made adaptable for outerwear use should be handled in the conventional procedures currently recommended and followed for woven goods. With regard to drycleaning and washability, the consumer will expect comparable or perhaps even better behavior from nonwoven fabrics. Still to be given a great deal of consideration are the many conceivable methods of finishing nonwoven fabrics, which may produce unique and novel effects for use in the apparel trade. Very attractive prints have already appeared, as well as dyed grounds, but little emphasis has been placed thus far on mechanical finishes which may be imparted to the nonwoven fabric. Such techniques should improve the acceptability of nonwoven fabrics in this field.

Nonwoven fabrics for various industrial uses may be considered as an entity depending upon the end-use requirements. Here again, however, where competition will be woven goods, economy, serviceability and utility must be made more obvious. It is estimated that nonwoven fabrics as a class will double or triple in volume every three years. Many factors, of course, could affect this in one manner or another. The influx of new processors of bonded fabrics is representative of the interest this commodity has aroused and of the possible growth pattern which is sure to follow. The over-all picture is extremely encouraging, and a considerable amount of research time of the manufacturer, chemical supplier and fiber producer is being spent in this direction. The combined efforts of the three parties working cooperatively should substantially increase the efficiency, productivity, merchandiseability and acceptability of nonwoven fabrics.

## REFERENCES


(1) Curlator Corp, East Rochester, N Y.
(2) Proctor and Schwartz, Inc, Philadelphia, Pa.
(3) Rodney Hunt Machinery Co, Orange, Mass.
(4) John Waldron Corp, New Brunswick, N J.
(5) Pellon Corp, Lowell, Mass.
(6) Trademark E I du Pont de Nemours & Co, Inc. Wilmington, Del.
(7) Permex Corp of America, Hawthorne, N J.
(8) Emery and Fuller, Merchandising Division, Textile Fibers Dept, E I du Pont de Nemours & Co, Inc, Wilmington, Del.

## Committee on Analytical Methods Report———

# ASSAY OF SODIUM HYDROSULFITE


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## I GENERAL

SODIUM hydrosulfite is of value in the textile industry as well as other industries because of its powerful reducing ability. When it performs useful work in reducing dyes, its divalent sulfur is oxidized to the tetravalent (sulfite) state. Any significant method of assay must then measure the amount of divalent sulfur, representing the reducing value of the hydrosulfite compound. The sample material is therefore titrated against some standard oxidizing agent.

The choice of oxidizing reagent is limited to those which are not affected by the impurities in commercial sodium hydrosulfites. Other limitations are imposed by the nature of the hydrosulfite itself. Hydrosulfite is rapidly and easily oxidized by air under certain conditions, particularly in the presence of heat, moisture and acidity.

The following three procedures, when faithfully applied, give precise and essentially comparable results:

a) The Ammoniacal Copper Method;
b) The Iodine Method;
c) The Rubine R Method.

The designation in each case indicates the identity of the oxidizing agent used. The methods are described in detail in Section III.

In its attempt to determine the most suitable analytical method for sodium hydrosulfite, the Committee reviewed the following methods:

Ammoniacal—Copper
Ammoniacal—Silver
Chromate
Ferric Alum
Hydrogen Peroxide
Indigo
Iodide—Iodate
Iodine-Formaldehyde
Methylene Blue
Polarograph
Potassium Ferricyanide
Potassium Mercuric Iodide
Potentiometric titrations
Rubine R

After considerable study the investigation narrowed itself down to three methods:

Ammoniacal—Copper
Iodine-Formaldehyde
Rubine R

These were tested by each member of the Committee and variations in results were discussed. Repeated experiments were made to test the efficiency of the revisions in the three methods. Finally the point was reached where it was possible to obtain reliable results by all three methods as revised.

Although these three methods give essentially comparable results, ***the iodine method is recommended because of its simplicity.*** The ammoniacal—copper and Rubine R methods should be used in rare cases where the hydrosulfite is known or suspected to contain significant amounts of thiosulfate, sulfide or other oxidizable impurity not inactivated by formaldehyde.

## II SAMPLING

The sampling of a container of sodium hydrosulfiite is of the utmost importance. In a drum or can of hydrosulfite, especially one which has been in storage for some time, the surface layer of hydrosulfite may well be weaker than the rest of the contents of the drum. When such a container is sampled, the surface layer should be pushed aside and the sample taken at a depth of 4-5 inches. The bottle in which the sample is to be put (usually a 4-oz bottle) is gently scooped into the hydrosulfite. The hydrosulfite is never poured into the sample bottle. Pouring promotes segregation of the coarser particles usually found in hydrosulfite.

The sample bottle taken should be three-quarters full and tightly sealed. It is obvious that a dry, clean bottle should be used.

Commercial hydrosulfite powders are usually quite heterogeneous, because they contain several components which tend to segregate because of different crystal sizes. Inattention to proper sampling procedure may result in assay errors of as much as four percent. Consequently, in addition to the precautions taken while filling the sample bottle, it is desirable to use as large a sample as feasible and to pay close attention to the following general principles in taking the sample for analysis:

> Stir and roll the sample well; then mix with a spatula (all in the sample bottle) just before removing the portion for analysis. The portion taken for analysis must not be removed by pouring from the bottle, but by spatula or by holding the sample bottle at a slight incline and pushing a weighing bottle into the "wall" of hydrosulfite until the weighing bottle is filled to the desired level. *Pouring* the sample from the bottle will result in nonrepresentative results because the portion thus secured for analysis will contain a disproportionately great amount of the larger granules of varying strength.
>
> When the analysis is to be performed immediately after the weighing, it is satisfactory to weigh the sample rapidly on an open balance pan. However, prolonged exposure to humid air should be avoided; so if the weighing operation is not rapid or if the actual analysis must be deferred after weighing, then the sample should be weighed in a closed dish.

## III ANALYTICAL PROCEDURES

### A) The Ammoniacal Copper Method

1) REAGENTS:—

*a) Standard Copper Solution*

This is a standardized solution of ammoniacal copper sulfate, used as the oxidizing agent in this method.

One liter is prepared as follows: Dissolve 50.0 grams of blue copper sulfate ($CuSO_4 \cdot 5H_2O$) in a mixture of 100 mls of concentrated ammonium hydroxide and 700 mls of water. Then dilute the solution to 1000 mls with water and allow to stand at least 24 hours. Remove any precipitate by filtering or siphoning. This solution retains its strength indefinitely if kept in a tightly stoppered bottle. Before use, its copper content must be determined accurately by an accepted

quantitative procedure, either the thiosulfate or the electrolytic method being preferred.

*Standardization by Thiosulfate.* Carefully pipet 20.00 mls of the copper sulfate solution into a 500-ml Erlenmeyer flask. Add about 50 mls of water; then set the flask on a hot plate and allow to simmer until all the blue color has faded out of the solution. Then add about 0.5 gram of sodium carbonate and 50 mls of water and heat again until the volume is approximately 25 mls. Add 10 mls of glacial acetic acid and warm until the brown CuO is all dissolved. Dilute to about 100 mls with water, cool to room temperature, add 5 grams of iodate-free potassium iodide, and titrate with a standard solution of N/10 sodium thiosulfate, using freshly made 1% starch solution as indicator. Titrate to the disappearance of the blue color, then add 1-2 grams of potassium or sodium thiocyanate and continue the titration with N/10 thiosulfate until the blue color disappears again. Record only the final buret reading as the amount of thiosulfate consumed. If B mls of N normal sodium thiosulfate is used,

$$\frac{B \times N}{20} = \text{normality of copper sulfate solution.}$$

*Standardization by Electrolytic Method.* Pipet 50 mls of the copper sulfate solution into a 300-ml tall form beaker. Add 50 mls of water, 100 mls of 10% (by volume) sulfuric acid, and 10.0 grams of ammonium nitrate. Then place the beaker on the electrolytic stand fitted with a rotating platinum anode and a tared platinum cathode. Electrolyze for an hour at 1.5-2.0 amperes and 3-4 volts with the anode rotating. Then wash down the walls of the beaker with a small amount of water and continue the electrolysis for about 30 minutes longer. With the current on, siphon off the acid liquor while adding water simultaneously so as to keep the plated copper immersed. Continue adding water thus until the electric current reads zero; then shut off the current, remove the cathode from the apparatus, wash it with ethyl or methyl alcohol, and dry it in an oven at 221-230° F (105°-110° C) for 15 minutes. Cool in a desiccator and weigh, to secure by difference the weight of copper contained in 50 mls of the copper sulfate solution.

$$\frac{\text{Wt of copper contained in 50 mls}}{3.177} = \text{normality of copper sulfate solution.}$$

*b) Carbon Dioxide*

It is necessary to displace the air from the titrating vessel with a stream of carbon dioxide which is free of oxygen. This carbon dioxide is best obtained from a cylinder of commercial carbon dioxide. To remove traces of oxygen which are frequently present, the $CO_2$ must be scrubbed through an oxygen-absorbing solution. A suitable scrubbing solution is conveniently prepared by dissolving one part of sodium hydrosulfite and two parts of sodium bicarbonate in twenty parts of water. This solution should be renewed every couple of days. Precautions should be taken to ensure that the delivery tube in the scrubber does not extend below the stopper so as to trap air in the scrubbing system. The delivery tube of the scrubber is connected through flexible rubber tubing to a glass gooseneck (made of 7 mm glass tubing), which extends into the Erlenmeyer titration flask to a point about 1¼ inches from the bottom of the flask. This flask is unstoppered, the gooseneck resting on the rim of the flask. When a titration is run, the stream of $CO_2$ should be regulated to such a rate that the bubbles appear in the scrubber just a little too fast to count. Then sufficient time must be allowed for the incoming $CO_2$ to displace the air completely from the flask before the titrant is added. As a check on the efficiency of the scrubber, it is well occasionally to let the $CO_2$ continue to run into the flask for about five minutes after the titration is completed. If the end point does not change, the $CO_2$ apparatus is functioning properly.

*c) Formaldehyde Solution, 37%*

2) PROCEDURE:—

The ammoniacal copper method has one outstanding advantage over the others only in cases where thiosulfate is a contaminant in the hydrosulfate. Neither thiosulfate nor any of the other common impurities in sodium hydrosulfite interferes with the applicability or accuracy of this method.

Mix 35 mls of 37% formaldehyde solution with 50 mls of water and add just enough sodium or potassium hydroxide to turn litmus paper blue (pH 8-9). Transfer about 75 mls of the mixture into a 100-ml volumetric flask by means of a long-stem funnel so that the neck of the flask is not wetted inside. Reserve the remaining 10 mls for later use.

Following the directions given in Section II, weigh accurately about 8 grams of sample. Pour this weighed sample as rapidly as possible through a short-stem funnel into the formaldehyde solution in the 100-ml flask, while twirling the flask so as to create optimum conditions for rapid conversion of the unstable hydrosulfite to the very stable sulfoxylate. (It is of the utmost importance to prevent the hydrosulfite from piling up in the flask during the addition, thus invariably giving low assay values.) Use the retained 10 mls of formaldehyde solution to wash down the funnel and the neck of the flask. Twirl until solution is complete, cool to room temperature, then dilute with water to 100 mls. Mix well, and pipet a 10-ml aliquot into a 125-ml Erlenmeyer flask for titration. Run a fairly rapid stream of carbon dioxide into the flask for at least half a minute before beginning the titration, and continue it throughout the entire titration. (Consult Section A-1-b above for details of the $CO_2$ addition.) Then, while holding the $CO_2$ gooseneck and the tongs in one hand, grip the neck of the flask with the tongs and proceed to run the standard copper sulfate solution into the flask from a buret *all at once* until within about 1-2 mls of the end point—without any heating. This will normally require about 40 mls of standard solution. Next, heat almost to boiling until the solution is essentially decolorized, then immediately continue the addition of the copper solution until the faintest perceptible blue color persists after 10 seconds of heating (at or near boiling). This is the end point.

*NOTE*: Do not boil until it is evident that the end point is very near. Prolonged boiling is not only unnecessary but is liable to result in a deficiency of ammonia and consequent inaccurate assay values. Carry out the titration as rapidly as possible, judging the end point against a white background under bright white light.

*Calculation:*

This is based upon the following equations:

a) $Na_2S_2O_4 + 2CH_2O + H_2O \longrightarrow NaHSO_2 \cdot CH_2O + NaHSO_3 \cdot CH_2O$

b) $NaHSO_2 \cdot CH_2O + 2Cu(NH_3)_4SO_4 + 2H_2O \longrightarrow (NH_4)_2SO_3 + Cu_2(NH_3)SO_4 + (NH_4)_2SO_4 + CH_2O + NaOH$

If B mls of N normal copper sulfate are used,

$$\frac{B \times N \times 8.705}{\text{Wt of sample in aliquot}} = \%\ Na_2S_2O_4$$

### B) The Iodimetric Method

1) PRINCIPLE—

The method is based on the fact that, when sodium sydrosulfite is added to an excess of formaldehyde, the sodium hydrosulfite couples with the formaldehyde to form the stable sodium formaldehyde sulfoxylate which may then be titrated with iodine. From the titration of the sulfoxylate with iodine, the percentage of $Na_2S_2O_4$ is readily calculated.

The reactions involved are:

a) $Na_2S_2O_4 + 2CH_2O + H_2O = NaHSO_2 \cdot CH_2O + NaHSO_2 \cdot CH_2O$

b) $NaHSO_2 \cdot CH_2O + 2I_2 + 2H_2O = NaHSO_4 + CH_2O + 4HI$

2) REAGENTS:—

*a) Iodine Solution, Approximately 0.1N*

Dissolve 18-20 grams of cp potassium iodide in about 300 ml distilled water. Transfer to this solution 12.8-13.0 grams of ground, cp resublimed iodine. Let stand with frequent shaking until the iodine has dissolved in the KI solution. Filter the solution through glass wool, washing well with water. Dilute the filtered iodine to one liter with distilled water. The solution thus prepared will be approximately tenth normal. Its standardization is discussed below.

Where numerous assays are run daily, it is very convenient to prepare several liters of iodine solution. Iodine solution should be kept in a tightly stoppered dark-colored bottle in a room where a fairly constant temperature may be maintained. On a very warm day, during the summer months when the temperature gets up into the nineties, the bottle containing the iodine reagent should definitely be kept at a lower temperature. The volatility of iodine reagent increases with rise in temperature and is definite at 90°F (32°C.)

*Standardization of Iodine Solution.* The iodine solution may be conveniently standardized against a sodium thiosulfate solution of known strength (approximately 0.1N).

Pipet out 50 ml of the thiosulfate solution and titrate with the iodine solution, using 1% starch indicator when the end point is approached.

The sodium thiosulfate solution is very conveniently standardized against 0.1N potassium dichromate. Dry at 221°F (105°C) for several hours some cp $K_2Cr_2O_7$ crystals. Cool in a desiccator. Weigh out 4.9035 grams of the dichromate, dissolve in water and make up to one liter. According to Griffin (*1*), $K_2Cr_7O_2$ can be obtained sufficiently pure to be used as a direct standard. The U S Bureau of Standards is a recommended source.

Pipet 50 ml of the dichromate solution into a 350-ml glass-stoppered flask; add 150 ml water and 5 ml conc $H_2SO_4$, and after cooling thoroughly, add 25 ml of 10% KI solution. Stopper the flask and let stand 10 minutes, then titrate the liberated iodine with the sodium thiosulfate. Use starch as indicator, but do not add it until near the end of the titration. The end point is denoted by the change in color of the solution from deep blue to light green.

*b) Formaldehyde Solution*

The formaldehyde solution is prepared by mixing 35 ml of fresh, clear 37% $CH_2O$ with 50 ml of distilled water. The resulting 35 ml of dilute $CH_2O$ will take care of one 8-gram sample of sodium hydrosulfite powder. It is very important that the pH value of the above mix be higher than 7, preferably 8-9. This pH may be obtained by dissolving caustic soda or sodium carbonate in the formaldehyde—water mix. Three tenths of a gram of sodium carbonate per 85 ml of formaldehyde mix gives a satisfactory pH of 9. The water used must be made oxygen-free by distilling, boiling or spraying with nitrogen.

Where numerous hydrosulfite samples are to be assayed daily, it is very convenient to prepare several liters of the pH-adjusted formaldehyde solution. Keep it in a dark bottle, and do not permit its temperature to get lower than 65°F (18°C) so as to avoid the danger of polymerization.

*c) Acetic Acid Solution*

Prepare a 20% aqueous solution from glacial acetic acid.

3) ANALYTICAL PROCEDURE:—

Carefully open the sample bottle and weigh out 8 grams of the hydrosulfite into a small, *dry, clean* weighing bottle, following the directions of Section II.

Measure out in a graduate 85 ml of the pH-adjusted formaldehyde solution. Pour about 75 ml of this into a 500-ml volumetric flask, holding the remainder in reserve. Pour it into the flask by means of a long stem funnel. When removing the funnel, do so carefully so as not to wet the neck of the flask. Now introduce another long stem funnel (dry) very carefully so as not to touch the sides of the flask neck. Grasp the upper neck of the flask as well as the funnel and start the formaldehyde in the flask in a circular motion, not violently. Pour into the funnel the 8 grams of hydrosulfite in a steady stream, but not fast enough to cause any appreciable accumulation of undissolved hydrosulfite. Continue swirling flask until all hydrosulfite has dissolved. Pour the balance of the formaldehyde in the graduate into the now-empty weighing bottle and pour it into the flask, rinsing several times with distilled water. Make the flask up to the mark with oxygen-free water and mix. The hydrosulphite is now "fixed" as the stable sulfoxylate.

Pipet 10 ml into a 250-ml beaker. Add 90 ml of oxygen-free water and 5 ml of the 20% acetic acid. Titrate with the tenth normal iodine, adding 1% starch indicator solution near the end point. The end point is sharp. Wait one minute before carefully reading the buret.

*Calculation:*

$$\frac{\text{ml iodine} \times N \times 0.0435 \times 100}{\text{Wt of sample}} = \%\ Na_2S_2O_4$$

With the iodine exactly 0.1N, and an 8.000-gram sample, the calculation becomes:

$$\text{ml iodine} \times 2.719 = \%\ Na_2S_2O_4$$

### C) The Rubine R Method

1) PRINCIPLE:—

A standard Pontacyl Rubine R (*CI* 179) solution is reduced with the sample. The excess dye is titrated with a standard titanous sulfate solution.

2) STATUS:—

The precision of the method at the 95% confidence level is ± 0.25% based on 3 single determinations by each of two technicians on each of 3 days (18 determinations, total). The Pontacyl Rubine R Method is suitable for routine control analyses of a large number of samples. About 10 minutes are required for a single determination.

3) REAGENTS:—

*a) Standard Titanous Sulfate Solution*

*Preparation of Solid $Ti_2(SO_4)_3$.* $Ti_2(SO_4)_3$ may be obtained commercially or it may be prepared as follows:

Place two liters of 20% $TiCl_3$ solution in a five-liter flask equipped with mechanical agitation. Sweep the flask continuously with a slow, steady stream of $CO_2$. Add 700 ml of conc $H_2SO_4$ from a dropping funnel in

about thirty minutes without cooling. Add one liter of methanol, the wash alcohol from a previous run, if available. Cool to 41°F (5°C), and filter on a Buchner funnel. Wash the precipitate on the funnel with five 200-ml portions of methanol keeping the washings separate from the main filtrate to be returned to the next run. Dry the product in a 122°F (50°C) vacuum oven and store in completely filled bottles for future use. Analyze the product by dissolving a 0.6 to 0.7-gram sample in 25 ml of distilled water plus 25 ml of 40% $H_2SO_4$ in a 300-ml Erlenmeyer flask, heating to the boil and titrating hot with standard 0.05 N ferric ammonium sulfate solution using methylene blue indicator.

*Calculation:*

$$\frac{\text{ml Fe soln} \times \text{N} \times 19.2}{\text{Weight of sample}} =$$

Reducing value calculated as % $Ti_2(SO_4)_3$

*Preparation of Standard $Ti_2(SO_4)_3$ Solution.* Weigh 50 grams of anhydrous tartaric acid for *each* liter of solution desired, dissolve in distilled water and dilute to volume. Add 48 grams of 100% $Ti_2(SO_4)_3$ for each liter and bubble $CO_2$ through the solution until the $Ti_2(SO_4)_3$ has dissolved. The standard $Ti_2(SO_4)_3$ solution must be set up and maintained in the same manner as a standard $TiCl_3$ solution.

*Standardization of Standard $Ti_2(SO_4)_3$ Solution.* Weigh 1.1-1.2 grams of 100% Metanil Yellow (*C I* 138) into a 300-ml Erlenmeyer flask. Add 50 ml of distilled water, 5 ml of 10% $Na_2CO_3$ solution, 50 ml of methanol, 25 ml of 25% sodium tartrate solution, and boil gently for five minutes while sweeping the flask with a slow, steady stream of $CO_2$. Titrate hot under $CO_2$ with the $Ti_2(SO_4)_3$ solution to the disappearance of the yellow color.

*Calculation:*

$$\frac{\text{Weight of Metanil Yellow}}{\text{ml } Ti_2(SO_4)_3 \times 0.09383} =$$

Normality of $Ti_2(SO_4)_3$

b) *Purified Du Pont Metanil Yellow*

Dissolve 400 grams of the dry, crude, unstandardized dye in one liter of boiling water, add 20 grams of Filter Cel and filter hot in a 6-inch Buchner funnel and flask previously heated to about 230°F (110°C). Transfer the filtrate to a covered beaker and heat, if necessary, until any crystallized dye has gone back into solution. Let cool spontaneously without agitation to room temperature and then continue the cooling in an ice bath, with occasional stirring to 41°F (5°C). Filter and wash with three 100-ml portions of cold ethanol. Dissolve the cake (without drying) in 900 ml of water and repeat the process, omitting the hot filtration. Dry this second cake at any temperature up to 212°F (100°C). The yield at this point, on the 100% basis, should be about 85% and the purity about 99.7%.

Treat about 75 grams of the dry cake with a liter of 2B alcohol, boil gently for 30 minutes and filter by gravity through a heated funnel and folded filter paper. Reheat the filtrate to dissolve any crystals that separated during filtration and let stand to cool spontaneously to room temperature. Continue the cooling to about 41°F (5°C), filter, wash once with cold ethanol. Dry at 212°F (100°C). The yield from this alcohol crystallization is rather poor, 50-60%, but the product is highly pure.

c) *Standard Pontacyl Rubine R Solution*

*Preparation of Pure Pontacyl Rubine R.* Dissolve 300 grams of the crude dye in 1200 ml of boiling water. Add 20 grams of Filter Cel, 20 grams of Darco, and 25 ml of glacial acetic acid. Filter through a double thickness of No. 1 Whatman filter paper in a 6-inch Buchner funnel and suction flask that have been preheated in an air oven to about 230°F (110°C). The filtration should be rapid and no crystals should deposit in the filtrate. If crystallization does occur, transfer to a beaker and heat until complete solution is restored. Pour the hot filtrate slowly, with constant stirring, into two liters of methanol heated on the steam bath to 122-140°F (50-60°C). Crystallization should not occur immediately. If it does, most of the product will be precipitated in a flocculent state, forming a thick, pasty mass that will not filter or wash well, and an inefficient purification will result. Cover the beaker and let stand for eight hours or more, without stirring, to cool spontaneously to room temperature. Pack the beaker in ice and continue the cooling, with occasional stirring, to 41°F (5°C). Filter on a 6-inch Buchner funnel and suck as dry as possible, pressing the cake down well. Wash with one 100-ml portion of cold 70% methanol, and two 100-ml portions of pure methanol. Without drying the cake, dissolve it in 1000 ml of boiling distilled water. Pour the hot solution into 200 ml of methanol as before, allow to crystallize, filter, and wash. Dry the final product at any temperature up to 212°F (100°C). To determine the suitability of the product for titration purposes, an end-point test is made as follows:

> Weigh roughly 2.5 grams of the dye and dissolve in 50 ml of distilled water. Add 5 ml of 10% $Na_2CO_3$ solution, 50 ml of methanol, and 25 ml of 25% sodium tartrate solution. Titrate with a 10% $Na_2S_2O_4$ solution, which has been made slightly alkaline with $Na_2CO_3$, to the color change from red to yellow. This final yellow color should not be darker than a 0.035% aqueous solution of $K_2Cr_2O_7$. If it is darker, the dye must be given an additional crystallization.

*Calculation:*

$$\frac{\text{ml } Ti_2(SO_4)_3 \times \text{N} \times 12.56}{\text{Weight of sample}} =$$

% Pontacyl Rubine R (mol wt = 502.4)

*Preparation of Standard Rubine R Solution.* Weigh 42 grams of 100% dye for *each* liter of solution desired and dissolve in distilled water. Dilute to the proper volume and mix well.

*Standardization of Standard Rubine R Solution.* Measure 25 ml of the standard dye solution into a 300-ml Erlenmeyer flask. Add 25 ml of distilled water, 5 ml of 10% $Na_2CO_3$ solution, 50 ml of methanol, and 25 ml of 25% sodium tartrate solution. Boil gently for five minutes while sweeping with $CO_2$. Titrate hot under $CO_2$ with the standard $Ti_2(SO_4)_3$ Solution.

*Calculation:*

$$\frac{\text{ml } Ti_2(SO_4)_3 \times \text{N}}{\text{ml Rubine R solution}} =$$

Normality of Rubine R solution

$$\frac{\text{ml dye solution}}{\text{ml } Ti_2(SO_4)_3} = \text{Relation R}$$

d) *10% $Na_2CO_3$ Solution*

e) *25% Sodium Tartrate Dihydrate*

f) *Methanol*

4) PROCEDURE:—

*a) Blank Determination*

The analysis of sodium hydrosulfite must be conducted at room temperature in order to avoid oxidation of the $Ti_2(SO_4)_3$ by sulfites present in the hydrosulfite. Dissolved oxygen present in the water used will react with $Ti_2(SO_4)_3$, so the analysis must be cor-

rected for oxygen present. Although this blank should be quite constant, it must be redetermined daily to insure accuracy.

Measure into each of two 300-ml Erlenmeyer flasks, 40 ml of distilled water, 5 ml of 10% $Na_2CO_3$ solution, 50 ml of methanol, 25 ml of 25% sodium tartrate solution, and from a buret, 10 ml of the standard dye solution. Boil one of the flasks gently for five minutes while sweeping with $CO_2$, then titrate hot under $CO_2$ with the standard $Ti_2(SO_4)_3$ solution. Titrate the other, also under $CO_2$ at room temperature, adding the $Ti_2(SO_4)_3$ solution very slowly near the end to avoid overtitration. To test this when the endpoint is reached, add one drop of dye solution. The red color should persist for at least five minutes.

Blank = ml for cold titration − ml for hot titration

*b) Analysis of Sodium Hydrosulfite*

Measure 50 ml of the standard Rubine R dye solution into a 300-ml Erlenmeyer flask. Add 5 ml of 10% $Na_2CO_3$ solution and then, in order, 50 ml of methanol and 25 ml of 25% sodium tartrate solution. Rapidly weigh a 1.3-gram sample of hydrosulfite in a weighing pan and transfer instantaneously in one portion to the above flask while swirling the flask contents with a steady circular motion. The swirl and the wetting action of the alcohol prevent caking of the hydrosulfite. Should caking occur, results are low. After the sample is completely in solution, sweep the flask with carbon dioxide for 30-60 seconds and titrate at room temperature with the standard $Ti_2(SO_4)_3$ solution. The color change at the end-point is from pink to amber and is quite sharp.

*Calculation:*

$$\frac{[(\text{ml dye} \times N_1) - ((\text{ml } Ti_2(SO_4)_3 - \text{blank}) \times N_2)] \times 8.706}{\text{Weight of sample}} = \% \ Na_2S_2O_4 \ (MW = 174.12)$$

or

$$\frac{[\text{ml dye} - R \ (\text{ml } Ti_2(SO_4)_3 - \text{blank})] \times N_1 \times 8.706}{\text{Weight of sample}} = \% \ Na_2S_2O_4 \ (MW = 174.12)$$

where:

$N_1$ = Normality of Rubine R solution
$N_2$ = Normality of $Ti_2(SO_4)_3$ solution
R = Ml dye equivalent to 1 ml $Ti_2(SO_4)_3$

## IV SUMMARY AND RECOMMENDATION

This report describes three analytical procedures for determining the reducing activity of sodium hydrosulfite. These methods are the iodine method, the ammoniacal copper method, and the Rubine R method.

Although these methods give essentially comparable results, the iodine method is recommended because of its simplicity. It is usable in all cases save where the hydrosulfite contains a significant amount of thiosulfate, sulfide or other oxidizable impurity not inactivated by formaldehyde. Trade experience has shown that these impurities rarely occur in significant amounts, and that they do not restrict general use of the method.

The ammoniacal copper and Rubine R methods are insensitive to these impurities, and may be used in cases where the hydrosulfite to be analyzed is known or suspected to contain them. The ammoniacal copper method is somewhat simpler than the Rubine R procedure as it does not involve the purification of dye standards. The Rubine R method might be considered a type of application test as it determines the reducing value of the hydrosulfite directly on a dye.

## V REFERENCE

(1) Griffin, R C, "Technical Methods of Analysis," 2nd edition.

# AATCC Calendar

COUNCIL
November (Hotel Statler, Boston, Mass)

NATIONAL CONVENTIONS
Nov 14-16, 1957 (Hotel Statler, Boston); Oct 30-Nov 2, 1958 (Hotel Conrad Hilton, Chicago); 1959 (Sheraton Park and Shoreham Hotels, Washington, DC); Oct 6-8, 1960 (Sheraton Hotel, Philadelphia, Pa); September 28-30, 1961 (Buffalo, NY)

DELAWARE VALLEY SECTION
Sept 27 (Joint meeting with New York Section at Hotel Hildebrecht, Trenton, NJ); Nov 1 (Kugler's Restaurant, Philadelphia, Pa); Dec 6 (Kugler's Restaraunt, Philadelphia, Pa)

HUDSON-MOHAWK SECTION
June 21 (Outing—Antlers Country Club, Amsterdam, NY)

MID-WEST SECTION
October 26 (Bismarck Hotel, Chicago, Ill)

NIAGARA FRONTIER SECTION
June 21 (Annual Outing—St Catherines, Ont); Sept 27 (Buffalo); Oct 25 (Joint meeting with CATCC Ontario Section at St Catherines, Ont); Dec 6, Feb 21 (Buffalo); Apr 4 (Joint meeting)

NEW YORK SECTION
June 21 (Outing—North Jersey Country Club, Wayne Twsp, NJ); Sept 27 (Joint meeting with Delaware Valley Section at Hotel Hildebrecht, Trenton, NJ)

NORTHERN NEW ENGLAND SECTION
Sept 13 (Outing—Wachusett Country Club, W Boylston, Mass); Dec 6 (Annual meeting, Hotel Continental, Cambridge, Mass)

PIEDMONT SECTION
Sept 27-28 (Annual Meeting—Hotel Barringer, Charlotte, NC)

RHODE ISLAND SECTION
Sept 27 (Ladies Night—Narragansett Hotel, Providence, RI); Oct 24 (Providence Engineering Society, Providence, RI); Dec 5 (Annual Meeting—Johnson's Hummocks Grill, Providence, RI)

SOUTH CENTRAL SECTION
June 21-22 (Outing—Lookout Mtn, Tenn); Sept 20 (Hotel Patton, Chattanooga, Tenn)

SOUTHEASTERN SECTION
Sept 14 (Harmony Club, Columbus, Ga); Dec 7 (Dinkler Plaza Hotel, Atlanta, Ga)

WESTERN NEW ENGLAND SECTION
Sept 27 (Rapp's Restaurant, Shelton Conn); Nov 1 (Annual meeting, Hartford, Conn. area); Dec 13 (Rapp's Restaurant, Shelton, Conn)

# Employment Register

57-9

*Education:* Northeastern Univ, BS, chemical engineering.
*Experience:* 20 years dye manufacturing—chief colorist; technical director; sales manager. All large firms. Excellent trade relationship.
*Age:* 46; married; references; resume on request.
*Position desired:* administrative—technical sales.
*Location desired:* open; now residing in Southeast.

57-10

*Education:* BS, textile chemistry, Philadelphia Textile Institute; MS, textile chemistry, Clemson A&M College.
*Experience:* Chemist, dyer-chemist.
*Age:* 26; references.
*Position desired:* Production research and development.
*Location desired:* South central USA.

# CHAPIN, SAMPSON ELECTED HONORARY MEMBERS

HAROLD C CHAPIN

ALBERT E SAMPSON

HAROLD Canning Chapin and Albert Edward Sampson, recently retired national secretary and treasurer, respectively, of the American Association of Textile Chemists and Colorists, have been elected unanimously to Honorary Membership in the Association by the 57 members of the Council.

Certificates attesting to their election to Honorary Membership in AATCC will be presented to Dr Chapin and Mr Sampson during the forthcoming 1957 National Convention at the Hotel Statler, Boston, Mass.

Only five other members of the Association have been raised to the level of honorary membership. William K Robbins, Walter E Hadley and Hugh Christison, all deceased; William R Moorhouse, treasurer emeritus of the Association, and William H Cady, past president and current editor of AATCC *Technical Manual and Year Book.*

Dr Chapin retired July 31, 1956, from his position as AATCC national secretary, a post he had held since 1933.

He was born September 20, 1883, in Gill, Mass, the son of Edward Sumner Chapin and Sarah (Canning) Chapin. He was educated at Cambridge (Mass) Latin School, and Harvard University, receiving his BA in 1904 and a PhD in 1910.

Dr Chapin was an instructor at Rensselaer Polytechnic Institute from 1909-1913; chemist and executive at National Carbon Co, 1913 to 1918; associate professor at Lafayette College, 1918 to 1920; and assistant professor and consultant, Lowell Textile Institute, 1920 to 1947.

He is a member of the American Chemical Society, the Chemists Club of New York, and the Harvard Club of Lowell, and is listed in *Chemical Who's Who.* In addition to his work as national secretary of AATCC, he was formerly secretary of the Northern New England Section, and held many important committee posts in the Association.

Dr Chapin married the former Pauline Elizabeth Durfee in September, 1917, and they have four children: Nancy Orne Chapin, Edward Canning Chapin, Elizabeth Durfee (Chapin) Heath and John Whitney Chapin. Dr and Mrs Chapin reside at 290 Pine Street, Lowell, Mass.

At the annual AATCC banquet in the Waldorf-Astoria Hotel last September, during the Perkin Centennial celebration, Dr Chapin was presented with a citation in honor of his retirement, which read in part:

"His 23 years of meritorious and inspiring stewardship as secretary of the AATCC brought dignity and international prestige to the Association; and broadened the horizons of its membership for the exercise of their talents and the progress of their industries."

* * *

Albert E Sampson, recently retired as resident manager of the Boston office of the National Aniline Division of Allied Chemical and Dye Corp is a charter member of the AATCC, having joined the organization at its founding in 1921.

He served AATCC in many capacities as a committee member and chairman and in advisory financial posts during the years he was a member of AATCC before becoming, in 1950, assistant national treasurer.

In 1954, he succeeded William R Moorhouse, now treasurer emeritus, as national treasurer of the Association, and held that post until his retirement from office at the end of 1956. Roland E Derby, president of Textile Aniline and Chemical Co, is now AATCC treasurer.

Mr Sampson was born May 24, 1892, in Gorham, Maine, and was educated in New England, taking his BS from the Massachusetts Institute of Technology in 1915.

Mr Sampson is married to the former Anna Lee. The Sampsons reside at 9 Thorndike Street, Beverly, Mass. He is a member of Rotary International, and the Necommen Society, in addition to his AATCC affiliation.

On the occasion of his retirement from the post of treasurer, the Council of AATCC passed a resolution of appreciation for the years of hard work he has given to that group.

Authorization for the election of Dr Chapin and Mr Sampson as honorary members is contained in Article IV Section 5 of the AATCC Constitution: "An Honorary Member shall be a person who has rendered distinguished service to the Textile Industry or to this Association." Election is by letter ballot of Council members, and an unanimous vote is required for election.

The National Headquarters Office
of the Association
Will Accept Until Further Notice

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for

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*of*

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IN FOUR VOLUMES

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The Society of Dyers and Colourists

*and*

The American Association of Textile Chemists and Colorists

at

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## EXAMPLE OF ENTRY IN PART I

**C.I. 69025** Anthraquinone **C.I. Vat Orange 1**

### APPLICATION

DYEING

METHOD
A/Q1 ☐
A/Q2 ■
A/Q3 ◘
Indigoid ☐

LEUCO COMPOUND
Alkaline, orange brown
Acid, brownish yellow

PROPERTIES (COTTON) Affinity and levelling, good. For data on strike, levelling and migration see *JSDC*, **66** (1950), 505
Suitable for pigment pad-jig, pad-steam, continuous piece and vat-acid processes

Vat in short liquor

**Cotton** Affinity and levelling good. Dead cotton covered fairly well

**Viscose** Good affinity at low temperature; at high temperature the hue is redder and duller

**Cotton/Viscose** Good solidity when dyed cold. At 50°C viscose dyed heavier than cotton

**Silk** Suitable

PRINTING

**Cellulose** Applied by normal processes giving redder and duller hues than when dyed. Dischargeable in pale and medium shades in the presence of an assistant

### FASTNESS PROPERTIES etc

| | A | B | C |
|---|---|---|---|
| CROSS DYEING (Chrome) ... ... | .. | 5 | 5 |
| HOT PRESSING ... | 5 | 5 | 5 |
| HYPOCHLORITE ... | 5 | 5 | 5 |
| LIGHT ¼–½ Normal | 5–6 | 6 | 6 |
| Normal | 6–7 | 6–7 | 6 |
| 2× Normal | .. | 7 | 6–7 |

| | A | B | C |
|---|---|---|---|
| MERCERISING ... | 5 | 5 | 5 |
| PEROXIDE BLEACHING | 5 | 5 | 5 |
| SODA BOIL (open kier) | 4–5 | 5 | 5 |
| SODA BOIL followed by HYPOCHLORITE ... (Combined test) | 4–5 | 4–5 | .. |

Tendering on exposure to light not accelerated

FASTNESS ON SILK (B) Degumming 5, Hot pressing 5, Light 6–7, Peroxide bleaching 4–5

### TEXTILE USAGE

DYEING

**Cellulose** On account of good level dyeing and all-round fastness as a shading colour where good light fastness is required. Suitable for curtains, hangings and for goods to be bleached, e.g. shirtings and towels

**Silk** For good fastness to degumming, peroxide bleaching and washing

**Unions** For cotton/viscose fabrics

PRINTING

**Cellulose** For direct print styles or as pigment with binder on cotton and viscose

HUE **Yellowish Orange**
ARTIFICIAL LIGHT: redder

| | |
|---|---|
| **Benzadone Gold Orange 3G ... ...** | **YDC** |
| **Caledon Gold Orange 3G ... ...** | **ICI** |
| **Carbanthrene Golden Orange 3G ...** | **NAC** |
| **Cibanone Golden Orange 3G ... ...** | **Ciba** |
| **Cibanone Golden Orange 3G℗ ...** | **Ciba** |
| **Fenanthren Golden Orange 3G ...** | **G** |
| **Indanthren Golden Orange 3G ...** | **FBy** |
| **Indanthrene Golden Orange 3GA ...** | **G** |
| **Indanthrene Golden Orange 3GF ...** | **G** |
| **Indanthrene Golden Orange 3GWP ...** | **G** |
| **Ponsol Golden Orange 3G ... ...** | **DuP** |
| **Ponsol Golden Orange 3GD ... ...** | **DuP** |
| **Sandothrene Golden Orange N3G ...** | **S** |
| **Solanthrene Orange 3J .. ...** | **Fran** |
| **Tinon Chlorine Golden Orange 3G ...** | **Gy** |

**Reactions on Cellulose**
$H_2SO_4$ conc.— greenish blue

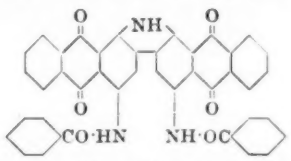
An example from the Vat dyes section of Part I is reproduced above and a specimen page (carbazole vat dyes) from the Anthraquinone section of Part II is shown opposite.

These two examples illustrate the information which appears in Parts I and II and demonstrates the dual reference system which connects the two Parts, e.g.

**C.I. Vat Orange 1** in Part I and **C.I. 69025** in Part II are complementary.

The format for the dyes in Part I varies with the amount of information available. Some dyes occupy a whole page, others only part of a page. For example, the space occupied by these notes may be used to accommodate information on **C.I. Vat Orange 2.**

**69005** **C.I. Vat Green 9** (*Brownish grey → Dull olive*)



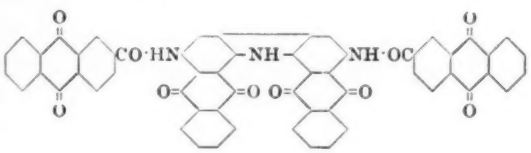
(*a*) Condense 1-benzamido-4-chloroanthraquinone with 1-amino-4-benzamidoanthraquinone in presence of copper and cyclise with chlorosulfonic acid
(*b*) Cyclise 4,4′-dibenzamido-1,1′-dianthrimide in sulfuric acid and oxidise the product

$Na_2S_2O_4$, alkaline — reddish brown; acid — yellowish brown

*Discoverer* — W. Mieg 1910
Bayer Co., *BP* 2702/09, 11932/09, 25986/09, 29352/10, 15753/12; *USP* 986521, 996109; *FP* 400653, 409407, 432449; *Sw.P* 55556; *GP* 220581, 225232, (*Fr.* **9**, 764, 1197), 228992, 239544, (*Fr.* **10**, 644, 638), 464292 (*Fr.* **16**, 1354)
Brit. Dye. Corp., *BP* 289191
*BIOS* 1493, 29; *FIAT* 1313, **2**, 135
*FIAT* 764 — Indanthrenoliv R
Vlies, *JSDC*, **30** (1914), 29
*Fierz-David*, 584 and Suppl. 82
*Houben*, 466, 662
*Mayer*, 181
*Fierz-David & Blangey*, Table 19
Fraser-Thomson, *JSDC*, **52** (1936), 242
*Thorpe*, **1**, 422

Soluble in *o*-chlorophenol
Slightly soluble in chloroform, pyridine
Insoluble in acetone, alcohol, toluene, xylene
$H_2SO_4$ conc. — red; on dilution — flocculent olive green ppt.

**69010** **C.I. Vat Green 22** (*Olive*)

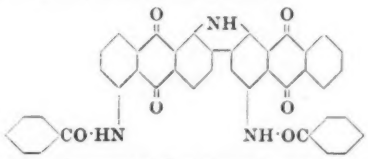


Condense 4,4′-diamino-1,1′-dianthrimide with 2 mol. 2-anthraquinonecarbonyl chloride and cyclise

*Discoverer* — F. Baumann 1929
I.G., *BP* 349714; *USP* 1819014; *FP* 696423; *GP* 513608 (*Fr.* **17**, 1398)
*BIOS* 1493, 28
*FIAT* 764 — Indanthrenoliv 3G

$H_2SO_4$ conc. — yellowish brown
$Na_2S_2O_4$, alkaline — reddish brown; acid — brownish yellow

**69015** **C.I. Vat Brown 5** (*Brown*)



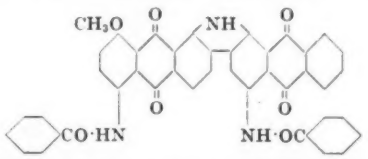
Condense 1-benzamido-5-chloroanthraquinone with 1-amino-4-benzamidoanthraquinone in presence of copper and cyclise with sulfuric acid

$Na_2S_2O_4$, alkaline — reddish brown; acid — yellowish brown

*Discoverer* — W. Mieg 1910
Bayer Co., *BP* 2702/09, 11932/09, 25986/09, 29138/09, 29352/10, 15753/12; *FP* 400653, 432449; *GP* 220581, 225232, (*Fr.* **9**, 764, 1197), 228992, 239544, (*Fr.* **10**, 644, 638), 491428 (*Fr.* **16**, 1349)
Brit. Dye. Corp., *BP* 289191
*BIOS* 1493, 10, 12; *FIAT* 1313, **2**, 106
*FIAT* 764 — Indanthrenbraun R, FFR
*Fierz-David*, 576 and Suppl. 82
*Mayer*, 181
Fraser-Thomson, *JSDC*, **52** (1936), 243
*Thorpe*, **1**, 422

Slightly soluble in xylene
$H_2SO_4$ conc. — dull wine red; on dilution — reddish brown flocculent ppt.

**69020** **C.I. Vat Brown 36** (*Reddish brown*)

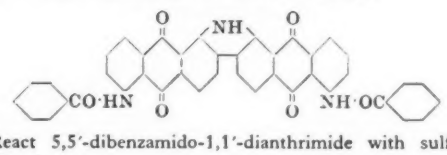


(*a*) Condense 1 - amino - 4 - benzamidoanthraquinone with 1 - benzamido-5-chloro-4-methoxyanthraquinone and cyclise
(*b*) Condense 1 - amino - 4 - benzamidoanthraquinone with 1 - benzamido-5-chloro-4-hydroxyanthraquinone, methylate the hydroxy group and cyclise with sulfuric acid

*Discoverer* — F. Baumann 1926
I.G., *BP* 298696; *USP* 1885172; *GP* 481362 (*Fr.* **16**, 1326)
*BIOS* 1493, 44
*FIAT* 764 — Indanthrenrotbraun 5RF
Fox, *JSDC*, **65** (1949), 513

$H_2SO_4$ conc. — violet; on dilution — red
$Na_2S_2O_4$, alkaline — brown; acid — brown (yellower)

**69025** **C.I. Vat Orange 1** (*Yellowish orange*)



(*a*) React 5,5′-dibenzamido-1,1′-dianthrimide with sulfuric acid and oxidise
(*b*) React 1-benzamido-5-chloro-anthraquinone with 1-amino-5-benzamidoanthraquinone in the presence of nitrobenzene and cupric chloride

*Discoverer* — P. Fischer 1910
Bayer Co., *Sw.P* 115114
Cassella Co., *GP* 239544 (*Fr.* **10**, 638), 249000 (*Fr.* **11**, 621)
*BIOS* 1493, 15, 77; *FIAT* 1313, **2**, 119
*FIAT* 764 — Indanthrengoldorange 3G
*Fierz-David* Suppl. 82
*Thorpe*, **1**, 423

Slightly soluble in nitrobenzene, tetralin, xylene
Insoluble in alcohol
$H_2SO_4$ conc. — greenish blue; on dilution — orange
$Na_2S_2O_4$, alkaline — yellowish brown; acid — yellow

# CONTENTS

**The work has been completely re-designed and brought up-to-date with the co-operation of all the dye manufacturers in Great Britain, America, Western Europe, India and Japan. The 2nd Edition is in three main parts.**

## PART I

**PART I**— consists of two volumes each of approximately 1,000 pages. It deals with all commercially homogeneous dyes and pigments in current use (approximately 4–5,000 dye entities and 25,000 commercial names) within groups relating to their main usage, e.g. Disperse, Acid, Azoic, Direct, Pigment, Solvent etc. The data included comprises the commercial name and manufacturer's initials, methods of application, fastness properties, established usages, reactions on the fibre and literature references relating to application. In the absence of a single accepted international system of fastness assessment it has been necessary, in this Edition, to make provision for the inclusion of data according to one or more of the following—

A— (American) The methods of The American Association of Textile Chemists and Colorists.
B— (British) The methods of The Society of Dyers and Colourists.
C— (Continental) The methods of the Deutsche Echtheitskommission.

An important feature of PART I is the sub-division of each usage group into hue groups— Yellow, Orange, Red, Violet, Blue, Green, Brown and Black. The hue classification has been made by reference to a Hue Indication Chart which has been designed specially for the purpose. A copy of the chart is included with each set of volumes.
Each dye or group of identical dyes carries a PART I serial name and number, e.g. **C.I. Vat Orange 1**, and also a five digit PART II number in those cases where the chemical constitution of the dye is known.


VOLUME 1 — Foreword — Preface — Contents of Volumes 1-4 — Introduction to Part I
Acid Dyes (including the premetallised dyes) — Mordant Dyes — Basic Dyes — Disperse Dyes — Natural Dyes and Pigments — Food (Food, Drug and Cosmetic) Dyes

VOLUME 2 — Direct Dyes — Sulphur Dyes — Vat Dyes — Ingrain Dyes — Azoic Dyes (sub-divided into Coupling Components, Diazo Components and Azoic Compositions) — Oxidation Bases — Pigments (inorganic and organic) — Solvent Dyes — Fluorescent Brightening Agents — Developers — Reducing Agents

## PART II

**PART II**— is one volume of approximately 700 pages. It contains data relating to chemical constitution, method of preparation, patent and literature references relating to manufacture or constitution, solubilities and reactions in substance for approximately 3,800 dyes and pigments. The 1st Edition recorded 1,316 constitutions, and this very big increase is partly due to the fact that many co-operating dye manufacturers have, for the first time, disclosed the constitution of certain of their dyes. In addition there is the information contained in the reports compiled by Allied Investigating Commissions on the German Dyestuff Industry.

The dyes are arranged in order based on their chemical constitution following broadly the lines of the 1st Edition. A number of refinements have been introduced and there are two new sections, viz— AZOIC (the coupling components and the diazo components for the production of Insoluble Azo dyes on the fibre) and PHTHALOCYANINE.

Each entry carries a five digit reference number. These numbers do not progress in unit steps as provision has been made to allow the insertion of new constitutions in later editions or supplements without having to change the numbers now allocated in the 2nd Edition. Each entry also carries the complementary PART I serial name and number thus providing easy cross reference to the data in PART I. It also contains alphabetical and empirical formula indexes to all the Intermediates relating to individual dye entities.

VOLUME 3 Introduction — Nitroso — Nitro — Azo — Azoic — Stilbene — Ketonimine — Triphenylmethane — Xanthene — Acridine — Quinoline — Methine — Thiazole — Indamine — Indophenol — Azine — Oxazine — Thiazine — Sulphur — Lactone — Aminoketone — Hydroxyketone — Anthraquinone — Indigoid/Thioindigoid — Phthalocyanine — Natural — Oxidation — Inorganic Pigments — Alphabetical index of Intermediates — Empirical formula index of Intermediates

## PART III

**PART III**— is one volume of approximately 400 pages containing the general index to **PART I** and **PART II** (see CONTENTS, [IV]). The Commercial Names Index includes the names (in italics) of dyes not in current use, many of which are related to data in PART I and/or PART II.

VOLUME 4 Hue Indication Chart (in pocket) — Dye Manufacturers' Initials (in alphabetical order) and Names — Dye Manufacturers' Names (in alphabetical order) and Initials — Abbreviations used in Parts I and II — Fastness Tests etc — Patents Index — Conversion Tables relating Colour Index 1st Edition to Colour Index 2nd Edition numbers and Schultz Farbstofftabellen (7th Edition) numbers to Colour Index 2nd Edition numbers and to AATCC Prototype numbers — Commercial Names Index — Additions and Corrections

# WOOD NAMED 14th OLNEY MEDALIST

P J WOOD, technical director of Royce Chemical Co and past president of the American Association of Textile Chemists & Colorists, has been named the 14th winner of the Olney Medal, highest AATCC award, given each year for "outstanding achievement in the field of textile chemistry."

Known professionally and to his many friends simply by the initials "P J," Mr Wood has had a long and distinguished career in business, as a professional man, and in various posts of service in community efforts.

Mr Wood, a charter member of AATCC, served as president in 1931 and 1932. By virtue of that service he is a permanent member of the group's governing Council. He is presently chairman of the AATCC Analytical Methods Committee, and a member of several other important committees, including Appropriations, Archives and the Technical Committee on Research.

The Medal itself will be formally presented to Mr Wood at the annual Olney Award Luncheon, to be held during the 1957 Convention at the Statler Hotel, Boston, Mass.

A summary of the Medalist's career, accomplishments and personal background will be made at the 1957 Olney Medal Award Luncheon by a close professional friend of the winner. This testimonial address will be made for Mr Wood by Milton Harris, director of research for the Gillette Co, and until recently president of Harris Research Laboratories, Washington.

Born June 21, 1880, in Leeds, England, Mr Wood was educated in English schools, receiving a diploma in chemistry and dyeing in 1898 from the University of Leeds.

Following graduation from Leeds, he served three years as an apprentice with William Grandage & Co, Bradford, England, and then returned to the University of Leeds as lecturer in the Department of Tinctorial Chemistry and Dyeing, for a period of two years.

He then came to the United States, and from 1903-1906 was chief chemist at American Silk Dyeing and Finishing Co, Hawthorne, N J, following

P J WOOD

which he became superintendent of Peerless Finishing Co, Nyack, N Y, until 1908.

From 1908 until 1934, he was vice-president and manager of Oriental Silk Printing Co, Haledon, NJ. After two years consulting work, he joined Royce Chemical Co, Carlton Hill, N J, as technical director, his present position.

In addition to his AATCC past presidency and charter membership, Mr Wood is also a life member of the Chemists Club, New York; fellow of the American Institute of Chemists; fellow of the Textile Institute (England); and fellow of the American Association for the Advancement of Science.

He has been secretary of the Passaic Valley Subsection of the American Chemical Society, and secretary of the Colorists Club for 15 years.

His service in civic and beneficial causes is extensive. He was Health Commissioner of the City of Paterson, N J, for five years; member of the board of managers of Passaic County Tuberculosis Sanatorium; president and director of the Haledon National Bank, for nine years; director of the Alexander Hamilton Garage; member of the board of managers, Paterson General Hospital, for 19 years; church-warden of the Trinity Protestant Episcopal Church, Paterson, for 30 years; and treasurer of the Passaic County Tuberculosis and Health Association, for 25 years.

Mr Wood has published 30 technical papers in *American Dyestuff Reporter*, including a number of translations from the French, German and Italian. He has also published papers and articles in a dozen other publications.

Mr Wood is married to the former Margaret May Davidson. They reside at 21 Emerson Avenue, Paterson, N J.

The Olney Medal is a heavy cast piece bearing a likeness of Dr Louis Atwell Olney, founder of the AATCC, on the front, and a simple inscription on the reverse side. The award is endowed by Howes Publishing Co, Inc, New York, and is awarded by an AATCC committee to any citizen of the USA as recognition for "outstanding achievement in the field of textile chemistry, including the development of chemical agents or chemical processes used in the manufacture of textiles, or methods for their evaluation."

Details of published works and professional qualifications of those proposed each year for the medal are usually contained in nominating briefs delivered to the Awards Committee. This Committee then considers those nominated and makes a final choice.

Under rules agreed to by the Association and Howes Publishing Co, Inc, the award must be made by unanimous decision of the committee of five, which includes: The Association president (ex-officio); a member of the faculty of an educational institution awarding chemical degrees; an employee of a concern manufacturing chemical products used in textile wet processing; an employee of a concern manufacturing textiles; a member at large of the Association.

Previous winners of the Olney Medal are:

1944—Louis A Olney
1945—Milton Harris
1946—William H Cady
1947—Edward R Schwarz
1948—Harold M Chase
1949—Charles A Seibert
1950—George L Royer
1951—Raymond W Jacoby
1952—Werner von Bergen
1953—Roland E Derby
1954—William D Appel
1955—Miles A Dahlen
1956—Walter J Hamburger

# 1957 CONVENTION NOTES

Exhibit space for the 1957 National Convention of AATCC, to be held at the Hotel Statler, Boston, Mass, Nov 14-16, is better than 50% purchased, it is reported. Information from exhibitors already contracted indicates one of the most diversified AATCC-sponsored exhibitions in recent years. In addition to dyestuffs, many new textile finishes, machinery and instruments will be on display.

* * * *

A partial list of participating exhibitors includes Alco Oil & Chemical Corp, Rohm & Haas Co, and Warwick Chemical Div of Sun Chemical Corp, who will display fabrics treated with their chemical finishes.

Important displays of dyestuffs, pigment printing colors and optical brighteners are planned by American Aniline Products, Inc, and The Hilton-Davis Chemical Co.

Goodyear Tire and Rubber Co expects heavy attendance at its booth, which will feature many new latex materials.

Machinery exhibitors are expected to participate heavily this year. A newcomer to AATCC Exhibits in this category is Elmo-International of North Bergen, NJ, who are the sole representative in America for Comerio Ercole SPA of Italy, one of Europe's leading manufacturers of textile equipment. Other machinery exhibitors include Bushman Products, Gaston County Dyeing Machine Co, Morton Machine Works and Smith, Drum & Co.

Instrumentation will be represented by Atlas Electric Devices Co, Custom Scientific Instruments Co, The Foxboro Co, Instron Engineering Corp, and the Macbeth Daylighting Corp.

Exhibits on salt and its applications in the textile dyeing and finishing industry are being planned by the International Salt Co, Inc and Morton Salt Co.

A partial list representing the textile publications and their services to the industry includes Howes Publishing Co, Inc (*American Dyestuff Reporter*), Fairchild Publications (*Daily News Record*), McGraw-Hill Publishing Co (*Textile World*), W C R Smith Publishing Co (*Textile Industries*).

Numbering among the exhibitors who have not released their exhibit plans are Union Carbide Chemicals Co, a Division of Union Carbide Corp; Fabric Research Laboratories, Inc; and the Tanatex Corp.

* * * *

Donald H Thomas, National Aniline Division, Allied Chemical and Dye Corp, chairman of the Exhibit Committee, has released a descriptive invitation brochure to prospective exhibitors for the Convention.

Richard R Frey, assistant secretary of AATCC and secretary of the Convention, advises that exhibitor applications for space are being received daily in Lowell and at the present rate a complete sell-out is indicated by late June or early July.

* * * *

Hotels Statler and Sheraton Plaza have made firm arrangements to accommodate Association guests and members attending the forthcoming National Convention.

Harrison M Gorton, Jr, American Aniline Products, Inc, chairman of the Hotel Committee, has announced that a priority system for assignment of rooms is being established to take care of the expected heavy registration.

# ACTIVITIES OF THE LOCAL SECTIONS

## South Central

THE South Central Section will hold its Summer Outing on June 21-22 at The Castle in the Clouds (formerly Lookout Mountain Hotel), Lookout Mountain, Tenn.

The outing will feature golf, horseshoe pitching and casting for the men; a luncheon and style show, swimming and sunbathing for the ladies; dancing and other entertainment.

John O Sweitzer, Scholler Bros, Inc, heads an outing committee of Paul O Anderson, Peerless Woolen Mills, finance; Lowell A Shive, ladies: Ed Jurczak, Burkart-Schier Chemical Co, golf; E E Burgner, Jr, Davenport Hosiery Mills, entertainment; Philip Stephenson, Bel Craft Chenilles, Inc, men's sports; E V Helms, Geigy Chemical Corp, prizes; William G Agnew, Dixie Mercerizing Co, registration; and L R Greenwood, Southern Dyestuff Corp, publicity.

## New York

AT the New York Section's final meeting of the 1956-57 season on May 17th, a resolution was passed stating that the present officers shall remain in their present capacities until January 1st, when nominations and elections shall take place. The motion was made in order that the New York Section would conform with its election of councilors, the national office, and the various other Sections.

It was stated that in August, a new set of laws and bylaws for the New York Section would be sent to all its members for approval.

As previously noted, the May 17th meeting was a ladies night affair, at which Sidney M Edelstein, president, Dexter Chemical Corp, spoke on "Strange and Humorous Stories of Our Past". Attendance at the affair, which featured a cocktail party and dancing, exceeded 150.

## Northern New England LTI Student Chapter

THE Northern New England Section met jointly with the Lowell Technological Institute Student Chapter on May 10, 1957 at the Institute campus at Lowell, Mass.

Tours of the Institute preceded the afternoon program, which consisted of a forum meeting on the subject of textile education. This featured 10-minute discussions by four senior panel members, followed by questions from a 4-man student panel.

Anthony J Villani served as moderator. Senior panelists were: Charles F Edlund, dean of faculty at LTI: John H Skinkle, professor of textile chemistry, LTI; Richard D Wells, Fabric Research Laboratories, Inc; and Arthur T Hartford, Jr,

(*Continued on page P456*)

E I du Pont de Nemours & Co, Inc. The student panel consisted of Louis Port, Robert H Keenan, Vernon H Ure, and John Santos.

Dinner was served in the LTI Cafeteria, following which the group moved to the Eames Hall Lounge for the evening program, under the direction of Leonard T Coppeta.

The following papers were presented by members of the LTI Student Chapter: "Dyeing of Hydrophobic Fibers" Alan A Denio and Therese Polak; "A Dyeing Study of Jute", Edward Landy; and "The Effect of Certain Acid Dye Structures on the Rate of Wool Dyeing", Edward W Makuch.

(L to r): John J Santos; Robert D Robinson, NNE vice chairman; Robert H Keenan; Edward W Makuch; Edward B Bell, NNE chairman; Alan A Denio, LTI Student Chapter chairman; George O Linberg, president, AATCC; Therese A Polak; Anthony J Villani; Prof John H Skinkle, LTI; and William W Pennock, NNE treasurer.

(L to r): George Lyna; William W Pennock, NNE treasurer; Edward Landy; Therese A Polak; Athena Letsou; Edward W Makuch; Maurice Seifer; and Robert D Robinson, NNE vice chairman.

(L to r): Prof John H Skinkle, LTI; Arthur T Hartford, E I du Pont de Nemours & Co, Inc; George P Paine, executive secretary, AATCC; Azel W Mack, NNE secretary; Anthony J Villani; Louis Port; Robert H Keenan; Vernon H Ure; and John J Santos.

Four LTI students received chemistry awards at the May 10th meeting. Left to right are Edward B Bell, NNE chairman; Alan A Denio, LTI Student Chapter chairman; George O Linberg, AATCC president, who presented the Association award to Robert C Schiek, next to him; Martin J Lydon, LTI president, who presented the Olney book prizes to Leonard Lifland, Maurice Seifer, and Robert Davis.

## Western New England

THE annual Ladies Light meeting of the Western New England Section was held on Friday, May 17th at Rapp's Paradise Inn, Ansonia, Conn, with approximately 80 members and guests in attendance.

The guest speaker of the evening was Miss Verne, nationally known beauty expert and director of education for Clairol, Inc. Miss Verne spoke on "Color and Personality". TV star Joyce Randolph ("Trixie Norton" to many Saturday night viewers), actress "Nicky" Greene and model Jo Ann Connelly aided Miss Verne in demonstrating some very distinctive hair styling.

Also on hand to entertain were members of the Society for the Preservation of Barbershop Quartet Singing.

1—TV star Joyce Randolph
2—Actress "Nicky" Greene
3—Joyce Randolph, "Nicky" Greene, and Jo Ann Connelly
4—Model Jo Ann Connelly
5—Miss Verne, Clairol School for Hair Dyeing, guest speaker
6—Al Rant, Laurel Soap Mfg Co, keeps the Clairol girls company
7—Tom Gillick, WNE chairman, and Mrs G indulge in terpsichore
8—A serenade by members of the SPBQS.
9—L to r: Victor DeMarco, Mr & Mrs Alexander H DeMarco, Shelton Hosiery Mills, Inc; John Hirn, National Aniline Div
10—Barber Shop Quartet
11—Mrs Bernard Lustig, Mr & Mrs George Alperin, Clairol Inc
12—L to r: John J Cervini, Cheney Bros; Mrs Carey; Mrs Cervini; Raymond J Carey, General Dyestuff Co
13—Andrew W Goodwin, Princeton Knitting Mills; Raymond Pollard, Warwick Chemical Co
14—Richard Eaton, UM&M; guest; Burton Blagman, Princeton Knitting Mills; Mrs Conrad Fleisher
15—Doris Goodwin, Al Rant, June Dickson
16—L to r: Mr & Mrs Paul W Reeves, Princeton Knitting Mills; Mr & Mrs Alfred A Caddell, Geigy Dyestuffs; Mr & Mrs Carmen T Verga, Housatonic Dyeing & Printing Co
17—L to r: Mr & Mrs Clinton W MacMullen, Olin Mathieson Chemical Corp; Mr & Mrs William H McCann, American Fabrics Co
18—L to r: Miss Verne, guest speaker; Tom Gillick, WNE chairman; Mrs Gillick; J Edward Lynn
19—Mr & Mrs Elmer A Ferrante, Housatonic Dyeing & Printing Co; Mr & Mrs George Alperin, Clairol Inc
20—L to r: Mrs Elmer Ferrante; Mr & Mrs Conrad Fleisher, Princeton Knitting Mills; Mr & Mrs C E Morrill, Princeton Knitting Mills

(Photos courtesy of A N Henschel)

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## Abstracts

*(Continued from page 432)*

resistant to rot, mildew, chemicals and burning.

For industrial uses the glass fabrics are usually given a special finishing treatment after weaving, to provide a bond between the fabric and the coating material employed. Several finishes are described by the author.

At present 14 mills are said to be weaving glass-fiber fabrics in appreciable quantities.—WHC

### A Critical Appraisal of the International Daylight Fastness Test

McLaren, K. *Soc Dyers Col* **73**, 121-7, April, 1957.

During the past eight years there has been much international discussion of the determination of the lightfastness of colored textiles, and now a method has been formally accepted by the International Organization for Standardization (ISO) which the author believes should eventually be adopted by most of the 37 countries which are members of ISO. In the United Kingdom the test has already been adopted as B B 1006:1955; it has not been adopted by the USA, however, as it would render invalid all the existing data obtained by using the AATCC sunlight test method and standards.

The International Daylight Fastness Test[1] has been critically examined to determine its reproducibility and its reliablity. Only one variable factor in exposure (the effective humidity) has been found to cause variations in assessment, and this is governed primarily by the proportion of sunlight falling on the pattern during exposure. This causes the assessments of humidity-sensitive materials to be up to two grades higher in sunny countries than in predominantly cloudy regions. This variation can be eliminated only at the expense of the reliability of the result, a solution which cannot be accepted. There is evidence to suggest, however, that the reproducibility of the method would be very good for the majority of fabrics, whose sensitivity to humidity, like that of the wool standards, is low.

The test is a reliable guide to the expected resistance to fading which will occur during use, even of sensitive materials, providing that the daylight test is carried out in the same country as that in which the fabric is to be used, or in one whose climate is similar, and providing that the fabric does not become wetted during use.

The author gives full details of the exposures, made all over the world, using the ISO daylight method, on which the above conclusions are based. He also presents a table listing the hours of actual *sunshine* per day recorded during the various exposures, the extremes being 1.9 hours in Moscow and 10.3 hours in Cairo.

Thirteen references to the literature are cited.—WHC

[1] A full description of the ISO Daylight Test may be found in the *Journal of the Society of Dyers & Colourists*, June, 1955, p 300-303. Essentially, it is in a continuous exposure under glass, day and night, regardless of weather or time of year, using eight dyed wool standards for comparison.

### The Lightfastness of Dyed Fibers —a Statistical Study

Giles, C H. *J Soc Dyers Col* **73**, 127-160, April, 1957.

This long and valuable paper is summarized by the author as follows:

About 16,000 published commercial lightfastness assessments have been examined statistically, and from the results the following general rules are formulated, which apply to most dyes and fibers:

a) On different fibers the fastness of a dye is higher, the higher the moisture regain of the fiber, provided that this is greater than about 4% (ie, the value for nylon). Thus dyes are usually faster to light on viscose rayon than on cotton, on cotton than on nylon, on wool than on silk, and on secondary cellulose acetate than on cellulose triacetate or nylon.

b) The above rule is reversed with fibers having regains below about 4%, ie, with the newer synthetic fibers; with these the fastness is usually higher on the fiber of lower regain.

c) Fastness is often reduced by i) solid foreign materials in the fiber, eg, dulling agents or the resin used in crease-resist finishes; or ii) either cationic surface-active agents or powerfully hydrogen-bonding compounds, eg, phenols or urea, used as dyeing assistants or aftertreating agents.

d) The increase in lightfastness with increase in depth of any dye is usually greater, the higher the regain of the fiber; it tends to be higher with dyes which are insolubilized in the fiber than with water-soluble ones. The increase in average fastness for a whole class of dyes for a tenfold increase in depth is usually about 1½ units for water-soluble, disperse and vat dyes, and about 3 units for azoic and sulfur dyes.

The effects summarized by these general principles are considered to be the result mainly of differences in the physical form of the dyes in the fibers, chemical action being important only in a few special cases, eg, the effect of titanium dioxide delustrant.

There is, however, a more general chemical influence which affects comparisons between certain fibers. This is the difference between the chemical nature of fading on protein and that on non-protein fibers (reduction and oxidation respectively) which has some effect on the comparative fastness of a given dye on fibers of the two classes, mainly at the upper and lower ends of the scale. Thus, dyes of low fastness on a nonprotein, eg, cotton, viscose rayon, cellulose acetate or nylon, are likely to have a fastness about one or two grades higher on a protein, eg, wool or silk (and perhaps Ardil fiber), and those of high fastness on a non-protein are likely to be graded one or two units lower on a protein.

Some suggestions are made of means by which lightfastness might be improved: eg, better fastness is more likely to be obtained by eliminating all small dye particles in the fiber than by encouraging larger ones to grow if small ones are also present. This is because the smaller particles tend to fade more rapidly than the larger, and therefore their fading rate determines the fastness grade of the dyed material, which is judged in the earlier stages of fading. Some types of dye molecule likely to exhibit high fastness are suggested.

The reliability of the fastness data (furnished by the dye firms) is discussed. Fastness assessments given by different manufacturers for the same dye appear to agree for wool dyes, but Continental assessments for dyes on cotton are usually about one unit higher than those given in this country. This is believed to be due to longer periods of sunshine on the Continent.

The paper concludes with 63 references to the literature.—WHC

### The Mechanics of Carrier Action in Dyeing Dacron Polyester Fiber

Schuler, M J. *Textile Research J* **27**, 352-8, May, 1957.

Dacron polyester fiber is usually dyed by two principal methods: either at temperatures above 100° C in pressure apparatus, or at temperatures of 100° or lower by adding organic compounds, called "carriers", to the dyebath to increase the dyeing rate.

In a search for carriers it was found empirically that many organic compounds increased the rate of dyeing. It is agreed by some that this increase in the rate of dyeing re-

sults from an increase in the rate of dye diffusion through the fiber. There is disagreement, however, on the mechanism by which this is accomplished. The author quotes five different authorities, each of whom has different ideas on the subject.

All of the work on the carrier dyeing of Dacron reported in the literature has been carried out in aqueous dyebaths. Since neither the dyes nor the carriers are usually completely soluble in water, it is difficult to specify their concentrations, and to compare them on an equal basis.

In the present investigation it was decided to use as the dyebath vehicle, instead of water, isooctane, which is an excellent solvent for both dyes and carriers. Two disperse dyes were employed, and several different carriers were compared. The dyebath temperature was 95°C.

The dyeing rate in isooctane was found to be only a fraction of the rate in water. However, by adding water to isooctane, a rate of dyeing was obtained, comparable with an aqueous system.

The author describes in detail the various tests, all carried out in a newly-developed apparatus, the Jackson Laboratory Kinetodyeograph, the construction of which is explained.

The effect of the carrier concentration on the diffusion of both dye and carrier into the Dacron fiber is illustrated by graphs.

The conclusion drawn from these tests are as follows:

The dyeing of Dacron polyester fiber with carriers and dyes proceeds by the following steps:

1) Equilibration of the surface layer of Dacron with organic carrier, water, and dye.

2) Diffusion of organic carrier and water into the fiber; this loosens the interchain forces.

3) Diffusion of dye through the somewhat more permeable fiber.

The above steps are concentration-dependent. All other things being equal, the greater the carrier concentration within the fiber, the more rapid the dye penetration. Carriers also assist the diffusion of other carriers.

The diffusion of dyes into the fiber depends on the activity of the dye in the system, and not on the concentration in any one phase.—WHC

### Factors Affecting Rate of Dyeing of Cationic Dyes on the Whiter, More Dyeable Orlon Type 42 Acrylic Fiber

Landerl, H P, Iannarone, J J, and Wilson, J J, *Dyes & Chemicals Tech. Bull (Du Pont)* 13, 9-15, March, 1957.

The production of Orlon type 42 acrylic fiber has been converted to a whiter, more dyeable merge that has a greater capacity for cationic (basic) dyes than previous type 42, and a faster rate of dyeing. It is now possible to obtain deep shades in less time, particularly when beck dyeing. (With disperse dyes, however, the dyeability of the new fiber is not appreciably different from that of the previous type 42).

The author recommends a temperature of 206° F and a dyebath pH of 5.0 for the Sevron (cationic) dyes, with additions of 6% Du Pont Retarder LAN, 10% anhydrous sodium sulfate, and a suitable surface-active agent. Dyeings made at lower temperatures exhaust more slowly.

The individual cationic dyes show a wide variation in their rates of dyeing. The time of half-dyeing of 18 of these dyes, at 206° F, listed in a table, varies from 7 to 32 minutes. However, mixing dyes with widely different exhaustion rates, which is sometimes necessary, can be done successfully, according to the author.

In general, the amount of Retarder LAN to be used will vary from 10% for very light shades to none at all for very heavy shades, when dyeing at 206° F, in order to obtain maximum levelness.

Curves are included, showing the exhaustion rate of Sevron Blue B at various temperatures from 200 to 212° F, and of 12 different dyes at 206° F; also the effect of varying the Retarder concentration on the rate of dyeing.—WHC

## FUTURE PAPERS

THE following papers have been accepted for publication in future issues of AMERICAN DYESTUFF REPORTER. Those papers which will appear in the *Proceedings of AATCC* are indicated by the letter (P).

"Changing Trends in Military Requirements"— V J MacLaughlin, Office of the Quartermaster General; T J Seery, Dept of the Navy; S J Kennedy, U S Quartermaster Corps; J H Ross, U S Air Force (P)

"Dyeing of Synthetic Fibers—A Dyer's Potpourri"—S G Turnbull, Jr, E I du Pont de Nemours & Co, Inc (P)

"Finishes and Blends Form the Future" —J A Woodruff, American Viscose Corp (P)

"Cleaning Synthetic Fur Pile Fabrics"— Dorothy Lyle, National Institute of Drycleaning (P)

"Dyeing of Orlon Acrylic Fiber—Application of Cationic Dyes"—J P Neary and R J Thomas, E I du Pont de Nemours & Co, Inc (P)

"Comparison of Wrinkle-resistant Finishes for Cotton"—T F Cooke, P B Roth, J M Salsbury, G Switlyk, and W J van Loo, American Cyanamid Co (P)

"How a New Dye is Evaluated to Decide if it is Worth Marketing"— A F Klein, American Cyanamid Co ( P)

"Fibers and Equipment Used in the Manufacture of Nonwoven Fabrics"— Wendell E Brennen, American Viscose Corp (P)

"Dyeing Blends of Dacron Polyester Fiber and Cotton"— J J Ianaronne, Jr, P L Speakman, O S Larson, R C Hurt, and E H Hinton, E I du Pont de Nemours & Co, Inc (P)

"Lighting Factors to be Considered in Textile Color Matching"—Warren B Reese, Macbeth Daylighting Corp (P)

"Practical Problems in Purchasing"— Harold F Jones, E I du Pont de Nemours & Co, Inc (P)

"Cost Accounting for Effective Control" —Franklin R Ehman, Hellwig Dyeing Corp (P)

"Practical Problems in Inventory Control"—Hans Halbach, Berkshire Knitting Mills (P)

"New Aspects of the Pad-Roll System" —Bertil Mellbin, Svenska Textilmaskinfabriken

"How to Get the Most Out of a Reducing Agent"

"Some Graphical Aids to Quantitative Reflectance Spectrophotometry of Dyeings"—Albert Darbey, Eastern Color & Chemical Co

"Selective Solvents for Analyzing Textile Fiber Mixtures"—Siegfried S Praeger, New York State Dept of Labor

"Use of Silicones in Wet Processing"— F L Dennett, Dow Corning Corp

"Absorption of Water by and Drying of Untreated, Laundered, and Used and Laundered Cotton and Linen Toweling" —Florence E Petzel, Oregon State Sollege

"Improvement of Heat Endurance of Cotton by Treatment with Dicyandiamide"—William G Sloan and Edmund M Buras, Jr, U S Dept of Agriculture

"Dispersed Growth Aeration of Cotton Finishing Wastes"— Nelson S Nemerow, North Carolina State College

"Characterization of Cellulose and Cellulose Derivatives by Means of X-ray Diffractometer"—L Segal and C M Conrad, U S Dept of Agriculture

"The Standard Depths of Shade in Relation to the Assessment of Colorfastness of Dyes on Textiles"—Paul Rabe, Farbenfabriken Bayer AG (P)

# News of the Trade

## SOCMA Spring Outing

Almost two hundred representatives of member companies of the Synthetic Organic Chemical Manufacturers Association met May 27-29 at Skytop, Pa, for the organization's annual spring outing.

Highlighted with activities progress reports by the Association's president, R W Hooker, and its various committee chairmen, the outing was also the scene of a banquet, addressed by Louis Fischer, internationally known journalist, foreign correspondent, and author. Mr Fischer spoke on "The New Era in Foreign Affairs."

## TRI Awarded New Contract

Textile Research Institute has been awarded a contract with the Agricultural Research Service of the U S Department of Agriculture to conduct research on cotton fiber properties. The project, entitled "Detailed Study of Cotton Fiber Extensibility and Its Practical Significance," involve an investigation of the fundamental principles of cotton fiber extensibility, its relation to the molecular structure of the cotton fiber, and its translation to the properties of more complex textile structures such as yarns.

The contract, which will run for a two-year period, is being administered by Thomas Kerr, fiber technologist of the Crops Research Division, Agricultural Research Service, USDA. Ludwig Rebenfeld of the TRI staff is in charge of the experimental work.

## CPDC Subsidiary Manufacturing Water-phase Textile Colors

Kent Printing Ink Corp, a subsidiary of Claremont Pigment Dispersion Corp, has commenced manufacturing water-phase textile colors at its new plant in Roslyn Heights, New York. A complete range of high-color-strength print pastes, suitable for application to a wide variety of commercial fabrics, is currently available from stock. Fabrics printed with Kentex colors under commercial conditions are claimed to exhibit superior washfastness as well as excellent wet and dry crock resistance.

## MIT Offering Course in Textile-Waste Disposal

Legislation for stream pollution abatement has placed demands upon the textile industry to provide a greater degree of purification of its organic industrial wastes. To review the recent advances in the fields of chemical and biological processing of textile wastes, the Massachusetts Institute of Technology is offering a one-week special summer program in Disposal of Industrial Wastes from the Textile Industry from Monday, August 9 through Friday, August 23.

This one-week program is intended primarily for textile and color chemists and chemical engineers who seek a better understanding of the role of biochemistry, both fundamental and applied, in processing organic chemical wastes. No formal education in biology is required of applicants, and no particular phase of the textile industry will be stressed. The presentation of waste treatment principles will lay the groundwork for application to cotton, wool, and synthetic fiber processing wastes; laboratory and pilot plant demonstrations will deal with wastes from each of these fields.

The Program will cover some of the fundamentals of sanitary chemistry and microbiology; mathematical and analytical techniques employed in waste treatment and stream pollution studies; the principles, design, and operation of physical, chemical, and biological waste treatment processes; and pilot plant studies.

The Program will be presented in two sessions from 9 to 12 and 1:30 to 4:30 Mondays through Fridays. Additional time will be available for discussions and plant visits. Tuition is $150, due and payable upon notification of admission. Academic credit is not offered. This Special Summer Program will be under the direction of Dr Rolf Eliassen, director of the Sedgwick Laboratories of Sanitary Science and Professor of Sanitary Engineering. Lectures, laboratory and pilot plant work will also be given by Dr Ross E McKinney, assistant professor of sanitary engineering and head of the Sanitary Microbiology Laboratory.

Further information and application blanks may be obtained by writing to: Office of the Summer Session, Room 7-103, MIT, Cambridge 39, Mass.

## General Calendar

**AMERICAN ASSOCIATION FOR THE ADVANCEMENT OF SCIENCE**
Gordon Research Conference on Textiles, July 8-12, Colby Jr College, New London, NH

**AMERICAN HOTEL ASSOCIATION**
June 24-25 (Hotel Textile Purchasing Course, Cornell Univ, Ithaca NY)

**AMERICAN OIL CHEMISTS SOCIETY**
Sept 30-Oct 2, 1957 (Fall meeting, Cincinnati); Apr 21-23, 1958 (Memphis); Oct 20-22, 1958 (Chicago); Spring, 1959 (New Orleans); Fall, 1959 (Los Angeles)

**AMERICAN SOCIETY FOR QUALITY CONTROL—TEXTILE DIVISION**
8th Annual Conference—Jan 30-Feb 1, Clemson House, Clemson, SC

**THE DRYSALTERS CLUB OF NEW ENGLAND**
June 21 (Annual Outing—Wachusett Country Club, West Boylston, Mass.)

**THE FIBER SOCIETY**
Sept 4-6 (Joint meeting with the Textile Institute—Hotel Statler, Boston, Mass; April 30—May 1 (The Clemson House, Clemson, SC)

**NATIONAL COTTON COUNCIL OF AMERICA**
October 2-3 (Chemical Finishing Conference—Statler Hotel, Washington, DC)

**SOUTHERN TEXTILE ASSOCIATION**
June 20-22 (Annual Convention—Ocean Forest Hotel, Myrtle Beach, SC)

**SYNTHETIC ORGANIC CHEMICAL MANUFACTURERS ASSOCIATION OF THE UNITED STATES**
Sept 10, Oct 8, Nov 12 (Palm Terrace Suite, Hotel Roosevelt, New York, NY)

## 50 Scholarships Available at LTI

More than 50 scholarships are available at Lowell Technological Institute for the coming year. In addition, numerous scholarship prizes, many of them amounting to tuition rates, will be offered throughout the year, in the areas of textiles, chemistry, plastics, paper, and leather.

All scholarships are granted by vote of the Institute scholarship and awards committee, and most are renewable provided satisfactory grades are maintained. Application may be made to the Director of Admissions, Lowell Technological Institute, Lowell, Mass.

Chemistry majors may compete for scholarships given by the Alliance Color and Chemical Corporation, the Interchemical Foundation, Ciba Company, Inc, and the Northern New England Section of the American Association of Textile Chemists and Colorists, which makes its award in memory of Ralph E Hale.

Textile scholarships are given by the American Viscose Corporation, Berkshire Hathaway, Inc, Burlington Industries Foundation, Caron Fine Spinning Associates, Mohawk Carpet Mills, Owens-Corning Fiberglas Corp, Shapiro Bros Factors Corp, Davis and Furber Co in memory of Russell Brown, Sylvan I Stroock, Inc, United Elastic Corp, Albany Felt Co, and the New England Knitted Outerwear Manufacturers' Association in memory of Samuel P Kaplan. The Arthur Besse, Gehring, and Ziskind memorial scholarships and the American Textile Machinery Association scholarship also are available to students in textile technology.

# • NEW PRODUCTS AND DEVELOPMENTS •

## Sherdye Plain Shades Now Dischargeable

The Sherwin-Williams Co, East Providence, RI, manufacturers of the Sherdye Padding Colors, which have been used as plain shades on most all fabrics, including blends, has made known its latest development for producing both white and colored discharge prints on these same pad-dyed grounds. Its is pointed out that, since the fastness properties of Sherdye colors are not adversely affected by resin aftertreatments, it is expected that this new approach to decoration will be welcomed.

Eight selected Sherdye self-shades are said to be completely dischargeable to a maximum white, including brilliant copper phthalocyanine blue and green, which are probably the fastest colors known to science. Blue G3W and Green GB3W require a slightly different discharging technique but not unconventional.

Five of these selected Sherdye self-shades are said to be color dischargeable with many vat colors. However, the Company points out that, since vat colors vary widely in fastness, it will be necessary to use discretion in their choice, to balance the peak performace of Sherdye Padding Colors, This is especially true where resin aftertreatments are involved.

Another group of Sherdye Padding Colors reportedly offers almost 100 percent resistance to any form of discharging. This provides for combinations of both discharge and reserve effects when the dischargeable and nondischargeable colors are used selectively for ground shades. In these instances, all colors in evidence will be the super-fast padding colors.

Aside from the various procedures possible and sometimes necessary, the suggested routine method of producing white and/or color discharge effects, is as follows: pad-dye, dry and cure the ground shades continuously, print, steam age, wash and finish. Some vat colors will require an oxidation after aging, and at this same point the copper phthalocyanine blues will need a short exposure to chemic. It is stated that both processes can be accomplished continuously in conjunction with soaping, however.

The Company points out the following features of the Sherdye Padding Colors: unsurpassed lightfastness, chlorine, acid and alkali resistance; excellent dryclean and AATCC #4 and #4A washfastness; completely satisfactory resistance to crocking; no chlorine retention; most other resins and functional finishing chemicals are compatable with Sherdye Colors, thereby permitting dyeing and resinating in the same operation; unlike dyestuffs, Sherdye Padding Colors are not substantive, which provides for dyeing small or large lots in virtually one continous shade from end-to-end; blended fabrics, including hydrophobic fibers blended with cellulosic types, can often be dyed in solid shades; many blends can be double-dyed for two-tone effects.

As quickly as research and development permits, The Sherwin-Williams Company will announce the availability of new dischargeable and reserve Sherdye Padding Colors.

## Lumicrease/Cuprofix Grey 3LBN paf

Lumicrease/Cuprofix Grey 3LBN paf is a new Sandoz direct dye, bluish grey in shade, which is said to offer the maximum fastness required for automotive and decorative fabrics.

Good wetfastness can be further improved by aftertreatment with Cuprofix, Sandofix and/or standard UF resin finishing, it is claimed. Lumicrease/Cuprofix Grey 3LBN paf is said to be stable to hard water. Level dyeing properties of this new color reportedly represent a great improvement over existing similar types. It is suitable for application by all conventional methods. Reserve of nylon and acetate is said to be exceptionally good.

A descriptive leaflet may be obtained from any District Office of Sandoz, Inc.

## Syntergents 35-K, 35-T

Nopco Chemical Company has developed two nonionic detergents, Syntergent 35-K and Syntergent 35-T, for the scouring of raw wool at low temperatures. Each product is designed to replace conventional scouring with alkalis or other nonionic detergents.

Syntergent 35-K is said to be applicable to all types of wool, but Syntergent 35-T is specifically suited to carpet wools. The advantage of eliminating the use of alkalis in the scouring liquor reportedly results in a softer, loftier, and whiter wool. Since the fibers retain their natural elasticity, the stocks are said to be easier to card, draft, and spin.

Additional advantages reportedly include low scoring temperatues of 120-130° F, and improved color of recovered wool grease.

For further information write to the Textile Chemicals Division, Nopco Chemical Company, Harrison, NJ.

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# • NAMES IN THE NEWS •

Allard

Keane

The appointments of **Edward J Allard** as resident manager for New England and **Michael E Keane** as assistant resident manager in the Boston Sales Branch of National Aniline Division, Allied Chemical & Dye Corporation, were announced recently. At the same time the retirement of **A E Sampson,** Boston resident manager, was announcd. In his new position, Mr Allard will report to **Harold L Rieg,** director of dyestuff sales and branch operations.

Mr Allard will make his headquarters in both the Boston and Providence offices. He has been with National Aniline since 1931, and has been resident manager of the Providence sales branch since 1950.

Mr Keane has been with National Aniline since 1921. He is being advanced from the position of Senior Salesman.

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**Kenneth H Klipstein,** general manager of American Cyanamid Company's Research Division, became vice president for personnel relations on June 1st.

**R P Parker,** assistant general manager of the Research Division, has been appointed manager of that division. **J F Bourland** will succeed Dr Parker as assistant general manager of the Research Division.

Mr Klipstein, who joined Cyanamid in 1933, has been responsible for the company's extensive research activities in Stamford, Conn, Bound Brook, NJ and Pearl River, NY for the past three years.

Dr Parker joined the Cyanamid organization in 1933 at Bound Brook, NJ, where he was named director of research in 1952. He was appointed assistant general manager of the Research Division in April, 1956.

Dr Bourland joined Cyanamid in 1941. He formerly held the post of technical director of the company's Engineering and Construction Division, to which he was appointed in May, 1956.

McLean

**Arthur F McLean** joined Bradford Dyeing Association (USA), Bradford, RI, as plant manager June 1.

Mr McLean, who has been associated with the textile industry for 20 years, formerly served as assistant manager of the Glenlyon Print Works, Phillipsdale, RI.

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**F C Ames,** formerly with Wiesner-Rapp Co, Inc, Buffalo, NY, has accepted a position as sales engineer with United States Supply Co, East Providence, RI.

The Company handles a complete line of wet-finishing and dyeing equipment and specialty supplies. Its district offices are in Red Bank, NJ and Rockingham, NC.

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**Carlyle G Morton,** laundry specialist, has been placed in charge of technical services to the laundry industry for the Silicate, Detergent, Calcium Division of Diamond Alkali Co, Cleveland, O. He will make his headquarters at the Diamond Research Center in Painesville, O.

Mr Morton has headed the Textile and Laundry Service Division of Swift & Co in Chicago since 1947. Prior to that time, he had been manager of the Textile and Washroom Research Department of the American Institute of Laundering.

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**Philip J Rogers** has been appointed technical representative for Kent Printing Ink Corp, Roslyn Heights, NY, manufacturers of water-phase textile printing colors.

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Werner G Smith, Inc, Cleveland, Ohio, has announced that **E W Kaufmann** will handle sales of its sperm oil and chemical products in the Philadelphia area.

Mr Kaufmann recently resigned as sales manager of the resin division of Archer-Daniels-Midland Company.

Nichols

Barber

A former director of research for Taylor Instrument Companies has rejoined the organization after an absence of several years. He is **Nathaniel B Nichols,** newly appointed to the position of chief engineer. He succeeds **Ralph E Clarridge,** who has been appointed to President Raymond E Olson's staff as specialist for new product lines.

The appointment of four men to supervisory positions in the Taylor's Application Engineering Department also has been announced. All the posts are newly created.

**John E Barber** becomes assistant department manager reporting to **George E Howard,** manager. Barber has been in application engineering ever since joining the firm in 1946.

Named as group leaders are **Elliott M Barr, Donald T Gregg and Henry Stoll.** Barr will handle the application side of electronic and electric contact instruments. In addition to several lines of control instruments, Gregg will head up instrument applications in the textile, power, food and rubber industries. Stoll will handle departmental responsibilities for the chemical, paper and petroleum industries.

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The vacancy in the position of superintendent of dyeing at the Eastern Division of the Franklin Process Company, which occurred at the death of **Kenneth L Harper** on May 9, 1957 has been filled by **David MacGregor.**

Mr MacGregor has been employed as chief chemist at the Philadelphia plant since February, 1935. He assumed his duties as superintendent of dyeing on May 27, 1957.

Prior to his employment with the Franklin Process Company, Mr MacGregor was associated with the Grasselli Division of E I du Pont de Nemours & Co, Inc.

# OBITUARY

### KENNETH L HARPER

Kenneth LeRoy Harper, 64, superintendent of dyeing at Franklin Process Co's Eastern Division plant, at Philadelphia, Pa, died suddenly at his home at Elkins Park on May 9th. He had held this position since 1922.

A graduate of Manlius Military School, Worcester Academy and Massachusetts Institute of Technology, Mr Harper was very highly regarded in the trade for his knowledge of dyes and their properties. During his years at Franklin Process Company, he was highly instrumental in the development and perfection of package dyeing. During recent years, he lent a great deal of his efforts to the successful development of processes for the dyeing of synthetic fibers in the package form.

Prior to his employment with Franklin Process, he was with the Eddystone Print Works.

He was a member of the American Association of Textile Chemists and Colorists, and the Technology Club of Philadelphia.

Mr Harper is survived by his widow, Caroline M; a son, James Barclay Harper of New York; and a brother, Carl R Harper of Marshalltown, Iowa.

### EDGAR R BEIGBEDER

Funeral services for Edgar R Beigbeder, a salesman for 24 years with National Aniline Div, Allied Chemical & Dye Corp, were held May 2nd at Pitman, NJ. He died April 29th at the age of 44.

Mr Beigbeder was a member of AATCC; the National Paint, Varnish and Lacquer Association, Inc; and Omicron Pi Fraternity. He was born in Boston and was graduated from the Lowell Textile Institute in 1933.

Mr Beigbeder was very active in civic affairs in the vicinity of Sewell, NJ. As a member of the Mantua Township Board of Education he served as chairman of the Pupils Progress Committee and was a member of the Personnel and Supply Committee. He was president of the Civic Association of Sewell; organist and choir director of the Pitman Presbyterian Church; a member of the Men's Association and Westminster Society of the Church; and assistant director of the Highland Male Chorus of Pitman. The latter group has designated its third Scholarship Award the "Edgar Beigbeder Memorial Scholarship" in his memory.

Mr Beigbeder is survived by his wife, Anna; four children; his parents; two sisters; and two brothers.

### ROBERT J ROSS

Robert J Ross, textile print blanket sales engineer for 10 years with the Textile Products Department, Dewey and Almy Chemical Company Division of W R Grace & Co, died May 23 after a short illness.

Mr Ross was a member of the Northern New Eugland Section of the American Association of Textile Chemists and Colorists.

Funeral services were held in Lexington, Massachusetts. Burial was at Shamokin, Pennsylvania. Ross, whose wife died recently, is survived by a son, James, 10.

### ADOLF WALZ

Adolf Walz, 70, partner in Sagamore Color & Chemical Co, Boston, Mass, and part owner of the Poughkeepsie Dyestuff Corp, Poughkeepsie, NY, died May 23, 1957 at the Mount Auburn Hospital, Cambridge, Mass.

Memorial services were held Saturday, May 25, 1957 at the Bigelow Chapel, Mount Auburn Cemetery, Cambridge.

A resident of Winchester, Mass, Mr Walz had been associated with the dyestuffs & textile industry since 1916. He was a member of the Drysalters Club of New England, and the Northern New England section of the American Association of Textile Chemists and Colorists.

He is survived by his widow, Gertrude C; two sons, Hans G, who was a partner with his father in Sagamore Color & Chemical Co, and Thomas; also two daughters, Ursula and Doris (Mrs Elwood Titus).

# TECHNICAL LITERATURE

**(It is understood that the following literature is available from the sources listed in italics).**

**THE ABSORPTIVE PROPERTIES OF BATH TOWELS**———*Henrietta M Thompson; Bulletin No. 19, May 1957, Department of Clothing, Textiles and Related Art, School of Home Economics, University of Alabama*———The absorptive properties of 11 white cotton bath towels, in a range of weights, purchased from local stores in Tuscaloosa, Alabama, in 1954 were studied after 1, 3, 5, 10, 15, 30, 45, 60, 80, and 100 launderings. They increased in amount and speed of absorption with successive launderings.

As the weight per square yard increased there was a corresponding increase in the quantity of water absorbed. However, the volume of water absorbed per gram of cotton was approximately the same for all towel weights through the forty-fifth laundering.

There was no significant relationship between price and such properties as fabric weight, thickness, loops per square inch, breaking strength or absorptive properties.

**EMULSION POLYMERS**———*Vikon Chemical Co, Elon College, NC*———New technical bulletins have been issued which describe the properties and uses of vinyl acetate emulsion polymers Vikon QL and Vikon RL and emulsion copolymer Vikon FPB.

Uses in various textile finishing operations are described, including cotton piece goods and cotton knit goods finishing, use in crease-resistant resin finishing and use in backing and coating textile fabrics.

The physical properties of the emulsions and the films are also described.

**GLUTARALDEHYDE (PENTANEDIOL)**———*Union Carbide Chemicals Co, Div of Union Carbide Corp, 30 East 42nd St, New York 17, NY*———Properties, shipping data, applications, reactions, and physiological properties are discussed in F40005A, a 4-page bulletin.

Glutaraldehyde is an intermediate for the manufacturer of textile and paper resins and corrosion inhibitors. It is an insolubilizing agent for both protein and polyhydroxy compounds.

Glutaraldehyde can be oxidized to glutaric acid, reduced to 1,5-pentanediol, and undergoes addition or condensation reactions. It undergoes most of the typical aldehyde reactions to form acetals, cyanohydrins, oximes, and hydrazones.

**INDUSTRIAL FILTER PAPER**——*Eaton-Dikeman Co, Mt Holly Springs,* Pa———Catalog #357, liberally illustrated with photographs, charts, and diagrams, presents a review of filtration practice, explains how paper, as a filtering media, helps solve problems in laboratory and industrial use, and is an aid in selecting one of the many grades of industrial filter paper for scores of applications.

Articles deal with such subjects as the separation of solids and fluids, industrial and laboratory filter papers, chromatography paper, folded circles for lab projects, bibulous paper, lens paper, a glossary of industrial filtration terms, etc.—*24 pages.*

**NONLUBRICATED BEARINGS MADE FROM CARBON GRAPHITE MATERIAL**———*Electro-Nite Carbon Co, 8601 Torresdale Ave, Philadelphia 36, Pa*———These bearings reportedly operate under very high temperatures, are chemically inert and have overcome many maintenance problems for industry. Carbon graphite bearings in operation are illustrated in ovens, process equipment, conveyors, etc. Also included are nomograph charts, grades & design information.—*16 pages.*

**SILICATE P's AND Q's**———*Philadelphia Quartz Co, 1158 Public Ledger Bldg, Philadelphia 6, Pa*———The May issue of "Silicate P's & Q's", Philadelphia Quartz Company's monthly house organ, mentions 22 widely accepted uses for the sodium and potassium silicates. Normally this sheet discusses just a few silicate applications, but in reference to "Chemical Progress Week", the significance of industrial chemicals to provide daily comfort and convenience in our modern way of life is emphasized. Some of the nation's most basic chemicals are mentioned as contributing heavily, both to this end and to our national economy.